JPRS-JST-88-027
9 DECEMBER 1988

FOREIGN BROADCAST INFORMATION SERVICE

JPRS Report

Science & Technology

Japan

JPRS-JST-88-027
9 DECEMBER 1988

SCIENCE & TECHNOLOGY

JAPAN

CONTENTS

/7310

New Ceramics Industry Update

43067137a Tokyo NEW CERAMICS in Japanese Jul 88 pp 82-86

[Article by Kazuo Utsumi of Mitsubishi Laboratories' Industrial Technology Department]

[Text] **Trend of Participation by New Ceramics Related Companies**[6]

Because the field of new ceramics has an extremely diversified range of applications, there are new participants from a variety of industries. Table 1 summarizes the motivations for entering the field of new ceramics, by industry. Companies in recession industries like steel and chemicals have taken up new ceramics as a part of diversification to escape economic stagnation, and other companies have had the primary goal of differentiating their products by giving them advanced capabilities. Both categories of companies have developed products while making the most of the technological potential they already have.

Among the kiln industries, the companies that deal with ceramics, those that succeeded with new ceramics relatively early were venture companies that dealt with electronic parts (packaging etc). These included Kyocera, Murata Manufacturing, and Narumi Ceramics. However, there are also later manufacturers that got their start in the kiln industries, such as Mitsubishi Mining & Cement and Chichibu Cement, that have taken up mass production of electronic parts. There are many companies that started with a great deal of knowhow regarding high-temperature refractories, and so were early entrants in the field of heat-resistant engineering ceramics. Manufacturers of fire-resistant substances like Kurosaki Refractories and Shinagawa Refractories, and glass makers like Asahi Glass are good examples of this.

Among chemical and textile manufacturers there are many which have taken advantage of their high potential in production technology, and have entered the market as raw materials suppliers. They supply many different types of materials; powdered raw materials for new ceramics have been supplied by Showa Denko, Denki Kagaku Kogyo, Toyo Soda Manufacturing, Ube Industries and others. Recently, however, there have been attempts to participate with parts and sinters for parts, not just raw materials. That is, there has been a shift to fields downstream. Sumitomo Chemical, for example, has long dealt with Al_2O_3, but has sold it outside the company only in powdered form.

Table 1. Motives for Participation in New Ceramics Activities

	Motive	Technical relationship	Contrast with existing projects, or viewpoint	Examples of future directions
Kiln industries	Improvement of basic line of business	Calcination technology; powder casting technology	Higher added value in basic line of business	Whole systems; advanced functions
Chemicals and textiles	Diversification; downstream penetration	Inorganic chemistry link (use of inorganic compounds in fine ceramics)	Better stance for dealing with market than relatively narrow field of fine chemicals	Refinement of raw materials; further downstream penetration
Steel and nonferrous metals	Home consumption; diversification	Refractories and grinding agents; accumulation of technology by producers and users	View that traditional element (steel) will not be completely replaced	Activation of subsidiaries
Electrical	Pursuit of advanced-function elements (upstream) (for home consumption)	User for electronic materials; technical capability as a user to evaluate, judge, and process with precision	Lack of knowledge of upstream (materials); impossibility of downstream development (of existing projects); necessity of targetted materials	In-house production; joint R&D with materials manufacturers
Machinery	Improved systems through introduction of new materials	Selection of more appropriate parts	Indispensability of upgrading perception function	Development of materials for various sensors
Automobiles	Reduced energy consumption; introduction of parts	Evaluation and use of catalyst materials; lengthy research on (energy saving) engine materials	Indispensability of R&D to achieve lighter, more efficient engines and new sources of motive power	Joint development with materials makers; development of new concepts based on new materials
Heavy industrial engineering	Improved products and systems through introduction of new materials	Ability to measure materials	Enhanced business opportunities through spread to related activities	Comprehensive fine ceramics systemization (involvement with related businesses)

Recently, however, it has begun development of sinters of ZrO_2 and SiC. Toray Industries has conducted product development, particularly of ZrO_2, and has put its energy into development and commercialization of sinters instead of just powders.

Steel makers had already established new materials divisions because of the slump in demand for steel materials, and had taken up new materials as an important theme of new projects. They have engaged in work on new ceramics as a part of that. Because large-scale facilities investment was directed into development of new materials instead of steel, the breadth of development targets and the speed of development have been spectacular. The most classic examples are Nippon Steel and Nippon Kokan. Many manufacturers of nonferrous metals and electrical cables have begun work on extensions of fields they have worked in for some time. Examples include the Al_2O_3 of Nippon Light Metal, the engineering ceramics that make use of Mitsubishi Metal's tool technology, as well as Sumitomo Electric Industries' BN and diamond. Others in this category, who have entered the market for electroceramics, are Mitsui Mining & Smelting (medium and high temperature thermistors) and Toho Zinc (high-purity hematite).

Manufacturers of electronic and electrical equipment are the biggest users of new ceramics now. But they, especially the integrated electrical machinery manufacturers, have come to develop and produce their own new ceramics in order to differentiate their products. They are actively moving upstream, just the opposite of the chemical companies. For example, Toshiba is making and selling sinters of Al_2O_3, AlN and Si_3N_4, as Hitachi does with sinters of SiC and SiAlON. They have another advantage in being able to take advantage of group companies (Toshiba Ceramics and Toshiba Tungaloy in the Toshiba group and Hitachi Metals, Hitachi Chemical, and Toyo Machinery and Metal in the Hitachi group).

Auto makers show promise as future users. At present, new ceramics have been introduced as engine peripheral parts, such as diesel engine glowplugs and turbocharger rotors, but the companies are engaged in joint development with materials makers to find materials suited to use in the engine itself. For example, Toyota has ties with Toshiba, Nissan with Hitachi and Nippon Tokyshu Togyo, and Isuzu with Kyocera.

Manufacturers of machinery have participated in a variety of ways. Many companies have just remained in the position of users. But some produce new ceramics products in subsidiaries, as does Hitachi Zosen, and some deal with new ceramics in ceramics related to their own products, as do Eagle Industry and Kubota. There are also companies like Mitsubishi Heavy Industries that have produced sinters, not to sell them to outsiders, but to acquire the knowhow to incorporate ceramics into their own products. There are, among camera manufacturers, many that have developed bioceramics; Asahi Optical and Nippon Kogaku are particularly noteworthy.

2. National Measures for Industrial Promotion[1,8]

MITI has devised the measures shown in Figure 4 in order to prepare a foundation for the new ceramics industry.

First, with regard to statistics on new ceramics products, the "Statistical Survey on Fine Ceramics Production" has been carried out since January 1986 under the Statistics Act, with the results published monthly in the MONTHLY REPORT OF MISCELLANEOUS STATISTICS.

Evaluation of new ceramics and standardization of testing methods are being carried out largely by the Japan Fine Ceramics Association. So far, the following four items have been made into Japan Industrial Standards:

(1) Bending strength test at normal temperature (JIS R-1601)
(2) Modulus of elasticity test (JIS R-1602)
(3) Chemical analysis of Si_3N_4 (JIS R-1603)
(4) Bending strength test at high temperatures (JIS R-1604)

There is to be a JIS for high-temperature modulus of elasticity tests soon, and those for high-temperature tensile strength and for fragility are scheduled for FY88.

Figure 4: Measures To Prepare a Foundation for the New Ceramics Industry[1]

The Fine Ceramics Center has responsibility for creating a database. In addition to documents and patent information, quantitative information on the basic characteristics of materials is to be put into a database.

In addition, important measures to promote international cooperation have been taken to an unprecedented extent. One of these, VAMAS (Versailles Project on Advanced Materials and Standards), is one of the international cooperation project on which agreement was reached at the 1982 Versailles summit; its purpose is to create standards to encourage, internationally, the practical application of new materials. Japan has participated in standardization research on tests for bending strength, hardness and wear.

In addition to these measures to prepare a foundation for production, there are also R&D projects in which the state plays a central role. These are included in MITI's budget for fine ceramics (first period of FY87) shown in Table 2. All together, this comes to more than ¥1.5 billion; it is evident that the government is deeply involved in efforts regarding new ceramics.

3. Characteristics of the New Ceramics Industry

Products that make use of new material are generally characterized by a high concentration of technology and high added value. They are said to be products with short life styles produced in small quantities with great variety.[9]

There is much trade in Al_2O_3; its unit price as a powder to be refined into aluminum ingots is about ¥0.035/gram. When it is graded by particle size and sold for use in fine ceramics, the unit price is from ¥0.35 to 0.5/g; when it is sold at an even higher purity for use in LSI substrates, the

Table 2: Overview of Budget Items Related to Fine Ceramics[a]

(unit: ¥1,000)

Item	FY86	FY87
Consumer Goods Industries Bureau		
1. Fine ceramics industry measures	14,854	14,015
(1) Fine ceramics industry trends survey	2,813	2,813
(2) Fine ceramics basic questions roundtable	3,437	3,458
(a) Ministry portion	2,768	2,791
(b) Trade and industry bureau portion	669	667
(3) Fine ceramics industry measures commissioned survey	8,604	7,744
(a) Material-specific and problem-specific research	2,933	2,933
(b) Fine ceramics international cooperation research	5,671	4,811
Other bureaus		
1. Fine ceramics standardization commissioned research	14,263	16,050
2. Fine ceramics (next generation)	972,256	1,201,586
3. Integrated regeneration system (Large project)	(1,071,590)	(2,122,514)
(Fine ceramics separation membrane)	193,600	
4. Very advanced processing system (Large project)	(20,000)	(1,099,331)
(Diamond coating)	--	
5. Fuel cell power technology (Moonlight)	(3,189,553)	(3,383,226)
(Cell materials)	(74,000)	(32,500)
6. Government/private joint research (laboratory portion)	(225,600)	(313,288)
(a) High-plasticity ceramics	28,947	undecided
(b) High-function medical materials	40,481	undecided
7. Conductive inorganic compound research (Consumer Industry subsidy)	80,636	78,880
8. Small business alternative energy R&D	(465,500)	(427,000)
(Ceramic heat exchanger for industrial furnace high-temperature exhaust gases)	33,350	undecided
9. Inspection-free facilities development and verification testing	(1,360,189)	(1,848,153)
(Light water reactor technology development, as a part of light water reactor improvement technology commissioned testing)	22,749	28,949
10. High-temperature corrosion-resistant petroleum production technology development	3,000	undecided
11. Preparation and promotion of database and information services	(66,000)	--
(Fine ceramics)	15,970	

price is from ¥5 to 8/g. Compared with the powder for refinery use, that is approximately a 200-fold increase. Thus a high degree of technology is required in the preparation of raw material powders, and differentiation of products has become difficult unless a company has a firm grasp of the needs of users. This trend is particularly obvious in the development of functional materials. Manufacturers who provide raw materials cannot meet needs by just supplying users with general products as they have in the past.

In such circumstances, a trend of business ties between companies involved with new ceramics has been apparent. Figure 5 illustrates the technical links between companies involved with new ceramics; there are many links between powder manufacturers or sinter manufacturers and users. As previously stated, this is a matter of powder manufacturers advancing into downstream fields and users advancing into upstream fields. The former style, under which raw materials were purchased and developed independently or made to the users' specifications by parts makers, is seen less frequently in this field. Consequently, the group of companies that deal with new ceramics forms a structure that fits in harmoniously with traditional manufacturers and users of materials; it is difficult to distinguish the "new materials industry" as specific companies, as has been done with the "materials industry."

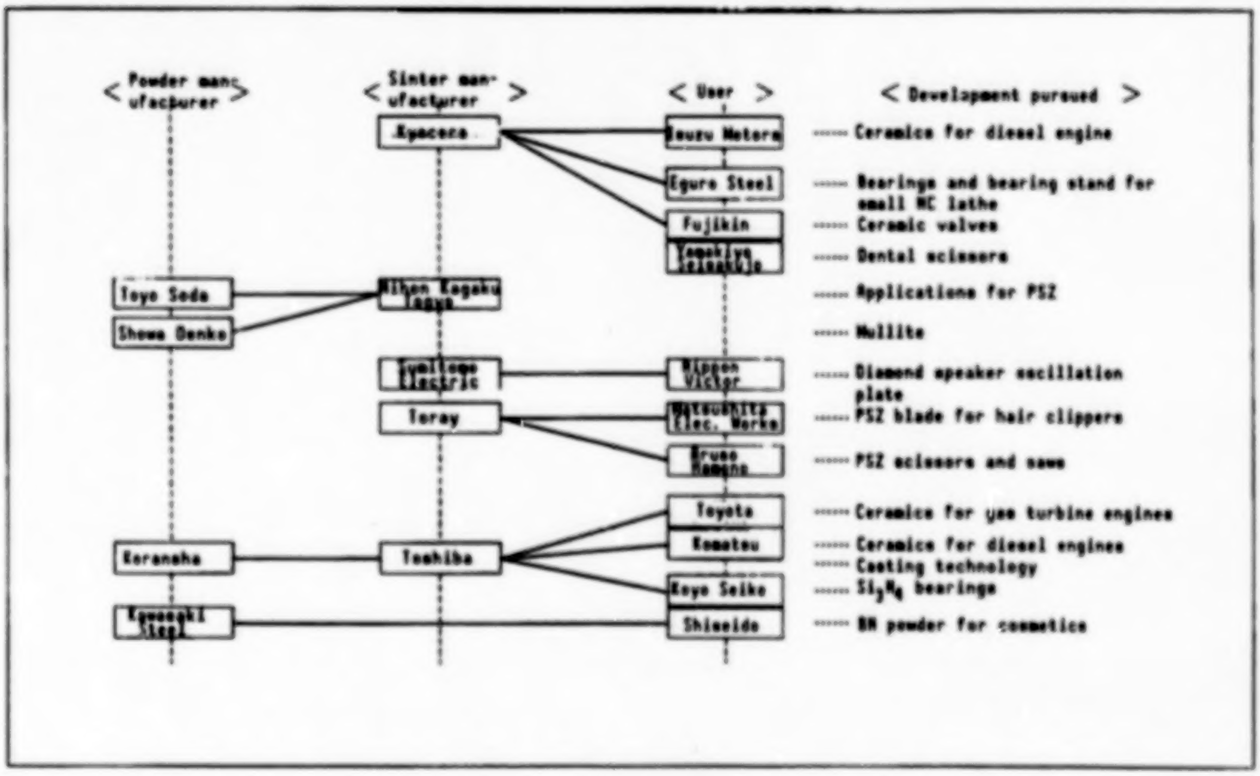


Figure 5. Trend of Business Ties Among Leading New Ceramics Companies

A large contribution to this comes from the shift of the new ceramics market in the direction of functional materials. New materials have come to make up a considerable portion of structural materials; if the method in which general ceramics are supplied to users had been followed, a group of companies like the present materials industry may have emerged. But since it is predicted that functional materials will remain as the leading element as of the year 2000, it is conceivable that the present "inter-industrial" structure of the new ceramics industry will continue a little longer.

4. Conclusion

As an industry, new ceramics is still in its infancy, but it is safe to say that new ceramics technology, or the preparation of an environment for the development of the new ceramics market, has gone quite far. For new ceramics to become independent as an industry, it will be necessary to develop ceramics as a structural material. At the least, it will be necessary for the scope of the market to grow to a level closer to that for functional materials. The government is aware of that, and is promoting the preparation of a climate for that. The trend of development of ceramics as structural materials will be watched closely.

References

1. MITI Consumer Goods Industries Bureau, Office of Fine Ceramics, ed.: "Fine Ceramics," Tsusan Sangyo Chosakai, 1987.

2. Akira Tsukamoto, et al.: "New Materials and the Development of Industrial Materials: Japan vs. the U.S. (conclusion)", NIKKEI NEW MATERIALS, No 28, 1 June 1987.

3. "Survey of Trends in the Fine Ceramics Industry," Japan Fine Ceramics Association, 1986.

4. "Survey of Trends in the Fine Ceramics Industry," Japan Fine Ceramics Association, 1987.

5. Masuo Sakubana, et al.: "The New Glass Industry and Raw Materials", KOGYO RARE METALS, No 93, 1988.

6. Special edition, "The Fine Ceramics Supply Map: How Do Manufacturers Deal With Them," NIKKEI NEW MATERIALS, No 18, 3 November 1986.

7. Eiko Kimura, "Fine Ceramics Industry Status and Prospects," Koshobo 1985.

8. Yasuo Tomita, "Japan's Fine Ceramics Policy and Future Course," CERAMICS, Vol 22, No 8, 1987.

9. "The New Materials Market and Company Strategies," TOSHI GEPPO (Mitsubishi Shintaku Toshi Komon), No 5, 1987.

9601/9635

Ceramics/Metal Bonding Technology Update

43067137b Tokyo NEW CERAMICS in Japanese Jul 88 pp 87-90

[Article by Kusuyata Shimamoto, director of Shimamoto International Patent Office]

[Text] 1. Closely Watched Bonding of Ceramics and Metal

New ceramics such as silicon carbide and silicon nitride have made their appearance, and have been put to practical use through technology for bonding with metals.

Technology for bonding ceramics and metals includes soldering (active metal and ultrasonic wave methods), solid-phase bonding (gas-metal eutectics, voltage stamping, friction pressure welding), fusion (electron beam), surface coating (thermal spraying, plating, CVD/PVD), and other methods (adhesives, mechanical bonding etc). As can be seen in the large number of patent applications to make practical use of ceramics, the advent of new technology is being watched from many quarters.

In terms of patent information, there are increasing numbers of patent applications in the area of technology for thermal bonding of ceramics and metal products (C 04 B 37/02).

The number of kokai patent cases laid open in 1980 was 33; it increased sharply from 30 in 1981 and 37 in 1982 to 72 in 1983, 132 in 1984, 169 in 1985, and 230 in 1986, then dropped to 179 in 1987.

By company grouping, the Toshiba group was far ahead with 110 cases, of which the Toshiba Corp. itself accounted for 104.

Next came the Hitachi group with 84 cases. Of these, 19 were from Hitachi Metals, 2 from Hitachi Chemical and 12 from Hitachi Zosen.

The Sumitomo group, especially Sumitomo Electric Industries, had 62 cases, as well as numerous combined applications that covered various fields.

The Toyota group had 31 cases, including 4 from Toyoda Central Research Institute, 6 from Aisin Seiki, and 3 from the Japan Automobile Parts Laboratories.

Table. Ceramic and Metal Thermal Bonding (C0 4 B 37/02) kokai patent cases

Organization/field	1983	1984	1985	1986	1987	Total
Agency for Industrial Science and Technology	4	1	3		8	
(AIST group)	6	10	15	4	35	
STA National Research Institute for Metals	1	3		4		
Government universities			1		2	3
Local public entities				1	3	4
Hitachi	5	3	12	12	8	40
(Hitachi group)	1	6	13	15	9	44
Toshiba	10	18	26	21	29	104
(Toshiba group)	3			3		6
Mitsubishi Heavy Industries	4	4	12	7	8	35
(Mitsubishi group)		1	1	5	3	10
Sumitomo Electric Industries	4	4	6	3	5	32
(Sumitomo group)		5	6	0	9	30
Toyota Motors	9		1		6	16
(Toyota group)		1	2	9	3	15
NGK Spark Plug	4	12	14	14	6	50
(NGK group)	6		9	6	2	22
Nissan Motors	2	11	4	4	3	24
(Nissan group)		2				2
Shipbuilding companies		23	3	3	3	32
Automobile related companies	1	7	5	14	13	40
Steel related companies	1				13	14
Ceramics companies	3	5	10	16	11	45
Chemical companies		1	3	4		7
Electrical machinery companies	4	3	7	13	3	30
Precision instruments companies	1	1	2		2	6
Machinery companies	3	1	7	9	9	29
Other companies	4	4	5	18	15	46
Individuals	2	1		3	2	8
Foreign companies	5	9	9	9	9	41
Total	72	132	169	230	179	782

The Mitsubishi Heavy Industries Group accounted for 45 cases, including 3 from Mitsubishi Electric, 2 from Mitsubishi Motors, and 2 from Mitsubishi Mining & Cement.

The following points appear in the kokai patents that have been noted.

2. Ultrasonic Wave Bonding by the Research Development Corp. of Japan (inventor: Shinichi Matsuoka)

The "ultrasonic wave method of bonding ceramics and metals" (kokai nr 61-14092 of 1986) developed by the Research Development Corp. of Japan

(28 June 1984) deals with a bonding method that uses ultrasonic wave oscillation. A metallic film is deposited on the surface of the ceramic; metal in contact with the metallic film is attached by pressure, and while under pressure is stamped on with ultrasonic wave oscillation. This bonds the metal and the ceramic. The same material is selected as the metal film to be deposited and the metal to be bonded.

The period for which the ultrasonic wave oscillation is applied, which is the time required for bonding, is in the range from 0.3 second to 3 seconds.

This invention can be used in a broad range of applications, including the fabrication of semiconductor elements, printed circuits and other parts.

3. Solid-phase Bonding by National Research Institute for Metals (inventors: Masayasu Kurahashi, Ikko Yoshiwara and Kazuyoshi Arai)

The "method of solid-phase bonding of alumina and metals or alloys containing Ti and O" [as published] (kokai nr 61-40877 of 1986) developed by the director of STA's National Research Institute for Metals (2 August 1984), deals with a solid-phase bonding method to firmly bond a metal or alloy to alumina without solder or the imposition of an intermediate layer. A metal or alloy that contains Ti and C, and which educes TiC on its surface when heated, is heated with alumina in an atmosphere that will not interfere with the eduction of TiC, under pressure and at a temperature at or above that at which TiC is educed.

Along with eliminating the defects of conventional methods in which an intermediate layer is interposed, this method enables solid-phase bonding of alumina and metals or alloys that cannot bond directly in solid phase.

4. Mechanical Engineering Laboratory's Thermal Bonding Method (inventors: Koichi Murakoshi, Masaharu Takahashi, Toshio Sano and Kenichi Murano)

The "method for thermal bonding of metals and ceramics" (kokai nr 61-141680 of 1986), developed by AIST's Mechanical Engineering Laboratory (11 December 1984), deals with a method of thermal bonding to connect metallic materials to ceramic bases, in the form of tubes or rods, by means of caulk processing. The metallic material is placed between the ceramic base and a coil that generates an electromagnetic force, and the metallic material is sealed to and bonded with the ceramic base by means of the electromagnetic force generated by passing a current through the coil.

When a large current is passed through the forming coil, an induced current flows through the outer surface of the flux concentrator, that is, through the outer surface facing the forming coil, and also along the surface of the slit to the surface of the cavity. This current induces a counter current in the surface of the metallic material; the repulsion between the current flowing through the metallic material and that flowing through the flux concentrator causes the simultaneous processing of four pieces of the magnetic material set into the cavity. By means of that processing, the metallic material is sealed and constricted into tubular form, and is bonded to the ceramic base.

The bonded ceramic base and metallic material are then removed from the magnetic forming equipment and placed in a thermal furnace where the bond is heated.

5. **Technology for Fixation of Ceramic Lining Material (inventor: Hiroshi Nomura of Hitachi Zosen)**

The "method for fixation of ceramic lining material" (kokai nr 61-228111 of 1986), developed by the High-reliability Marine Propulsion Plant Technology Research Group (29 March 1985) deals with a method for fixing ceramic linings on piston tops or inside cylinder heads in low-speed, two-cycle diesel engines. To fix ceramic lining material to the inner surface of a metallic base to form a chamber for high-temperature gases, the exposed surface is fixed in place with a pin in a position that will not come into direct contact with the hot gases. This pin is composed of a threaded rod, which screws into the metal base, and a head that contacts the ceramic lining material through a number of spring rings and also fits into a slide and indentation forming a clearance between the spring rings, such that the difference in thermal expansion between the metal base and the ceramic lining is absorbed by the indentation, slide and clearance.

6. **NGK Insulators' Ceramic-Metal Bond (inventors: Tsutomu Oda and Takao Soba)**

The "ceramic-metal bond" (kokai nr 62-4528 of 1987), developed by NGK Insulators (10 July 1985), deals with a ceramic-metal bond in which the ceramic and metal are joined by physical means. Convexities in the ceramic part fit into concavities in (or holes through) the metal part to form a ceramic-metal bond. The bond is characterized by the point that, when the convexity of the ceramic part is removed from the concavity of the metal part, the difference between the outer diameter of the convexity and the diameter of the concavity (after removal) is at least 0.2 percent of the outer diameter of the ceramic convexity.

7. **Toyota's Ceramic Rotor (inventor: Yoshihiko Tsuzuki)**

The "ceramic rotor" (kokai nr 62-13702), developed by Toyota Motors (10 July 1985), deals with a ceramic rotor having a spindle and a disk, and particularly the structure of the bond between the spindle and disk.

This technology consists of a ceramic rotor made up of a spindle and a disk fixed at the end of the spindle that rotates together with the spindle. The spindle and disk are connected by means of a metal torque transmitter that meshes with slots in both the spindle and disk, such that no relative motion is possible. The torque transmitter is subjected to a plastic deformation that causes a tight seal between the spindle and the disk. The metal filling the bottom portion of the slots is fixed by welding.

This invention makes it possible to bring to a practical level the previously unrealized unitary bond between a ceramic spindle and disk.

8. Inorganic Institute's Apatite Complex (inventor: Kazuaki Hirota)

The "apatite complex production method" (Kokai nr 62-27379 of 1987), developed by the STA's National Institute for Research in Inorganic Materials (26 July 1985), is a method of producing apatite sinter complexes that are useful for implants in artificial bones or teeth. The technology consists of production of complexes obtained by bonding apatite and another material. In this process, the material to be bonded is placed in contact with the apatite in an airtight container. After the air has been exhausted, the material and the apatite are made to adhere by adding pressure evenly using a fluid from outside the container as a pressure medium. Then the temperature and pressure from outside the container are raised or lowered while being maintained within a given range of values.

9. Toshiba's Method of Bonding Ti and Ceramics (inventors: Shoko Nakahashi, Hiroaki Takeda and Makoto Shirakane)

The "Ti and ceramics bonding method" (kokai nr 62-46975 of 1987), developed by Toshiba Corp. (23 August 1985), deals with a method of bonding silicon nitride ceramics and Ti or Ti alloys. The method firmly bonds titanium with the silicon nitride ceramic at the low annealing temperature of titanium.

This technology consists of applying to the surface of the silicon nitride ceramic a metal that is highly reactive with silicon, and heating the ceramic and metal in a vacuum. The nitrogen and silicon at the surface of the silicon nitride ceramic are thus separated, and the surface of the silicon nitride ceramic is metallized by the reaction product of the newly available silicon and the metal or alloy. After Al or an Al alloy is applied to the junction between the metallized layer and the Ti or Ti alloy, the junction is heated to a temperature at least as high as the melting point of the Al or Al alloy.

10. Toyota's Technology for Bonding through a Thermal Stress Buffer (inventors: Tamio Shinozawa, Masahiro Ogawa, and Noriaki Nishino)

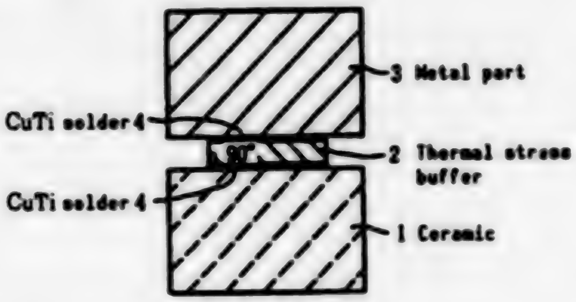
The "method of bonding ceramic and metal parts" (Patent Application Laid-Open #56381 of 1987), developed by Toyota Motors (5 September 1985), deals with a method of bonding ceramic and metal parts through a thermal stress buffer. The area of the bond between the ceramic part and the thermal stress buffer is greater than that of the bond between the metal part and the thermal stress buffer, and the angle between the edge of the thermal stress buffer and its bond with the ceramic part is at least 90 degrees.

This bonding method can be used in such applications as bonding pads to automobile camshafts or bonding gas turbine shafts to other shafts.

Placing a thermal stress buffer between the ceramic part and the metal part greatly reduces cracking caused by thermal stress and greatly increases the strength of the bond.

Making the angle between the edge of the thermal stress buffer and its bond with the ceramic part at least 90 degrees greatly increases the strength of the bond.

The figure is a structural diagram that shows the main points of this method of bonding ceramic and metal parts:



[Figure]

11. Honda Motors' Solder Technology

The "method of bonding ceramic rotors to metal spindles" (kokai nr 62-70275 of 1987), developed by Honda Motors, deals with a method of bonding ceramic rotors, such as ceramic turbine foils, to metal spindles.

The technology consists of bonding the ceramic rotor and the metal spindle with solder while the end of the spindle is engaged with the axis of the rotor. The spindle is engaged with the axis of the rotor using a structurally separate metal sleeve; after the sleeve is fitted to the axis of the rotor and soldered, it is bonded to the metal spindle by means of electron beam welding.

9601/9365

Application of Retoractory Metals for Ultra-LSI's

43067610 Tokyo KINO ZAIRYO in Japanese Aug 88 pp 12-19

[Article by Naoki Yamamoto, Central Research Institute of Hitachi, Ltd.: "Applications of Metals With High Melting Points to Super-LSI"]

[Text] Advances in the levels of integration of LSI's have reinforced the importance of metals with high melting points as new materials. Polycides (silicides/polycrystalline Si) are important as materials for manufacturing gate electrodes for LSI's ranging from 256 Kbits to megabits. Great expectations are placed on W and Mo as materials for low-resistance electrodes for ultra-LSI's 64 Mbits or above. In this paper the author describes the material properties that are important for developing these LSI processes.

1. Foreword

Among the major technologies that have contributed to the rapid growth of MOSLSI are the growth technology for high-quality SiO_2 films, the technology of fabricating no-defect Si wafers and photolithography technology. Of all of these, the greatest motive force has been the application of polycrystalline silicon (poly-Si) to gate electrodes (Table 1).

The emergence of the poly-Si gate electrode has completely solved the problem of unstable MOS characteristics--a problem associated with the earlier MOS devices that used an Ai gate. In particular, variations in characteristics caused by contamination from movable ions from the alkaline metal impurities found inside SiO_2 can be reduced to levels below the limits of detection by high sensitivity electric evaluation technology. However, as miniaturization of the elements in the wake of increasingly higher integration of LSI's advances, it will no longer be possible to ignore signal delays caused by resistance in the poly-Si electrodes and wiring. Therefore, in VLSI's--following the 256K D-RAM--Si compounds (silicides) with high melting points have come to be widely used for the gate electrodes and in the wiring. Metals with high melting points have been adapted in various ways to MOSLSI technology, but here the author concentrates on the application of metals with high melting points to gate electrodes and wiring, a theme I have been studying.

Table 1. Development of MOS·LSI and Advances in Major Technologies

Item \ Year	1965	1970	1975	1980	1985	1990	1995
D-RAM integration		1Kb 4Kb	16Kb 64Kb	256Kb	1Mb 4Mb	16Mb	64Mb
Used MOS elements	P channel MOS		n channel MOS		C-MOS		
Gate electrode	Al gate		Poly-Si gate		Polycide gate		High melting point metal gate
Number of elements in a memory cell		3-transistor		1-transistor + 1-capacitor			SOI + 3-transistor
Diffusion layer forming method	Thermal diffusion			Ion implantation			Micro-ion beam
Lithography technology		Photoexposure (contact aligner)			Photoexposure (projection)	Electron beam exposure	X-ray exposure
Processing technology		Wet etching		Plasma	Reactive sputtering		
Design rule (microns)		10 7	5 3	2	1.3 0.8	0.5	0.3

2. Electrodes, Wiring and the Law of Proportional Contractions

Scaling down the measurements of the various segments of a MOS transistor proportionally will drastically improve the device's performance, as illustrated in Table 2. Miniaturization in accordance with the law of proportional contractions does not give rise to a signal delay in the unit block of an LSI, as shown in Figure 1. But as the total length of the wiring between unit blocks increases thanks to increasingly finer wiring and increasing LSI integration, there occur a conspicuous increase in signal delay in the wiring as well as a conspicuous drop in voltage and an increase in current density (Table 3). Consequently, to reduce the effects of those problems associated with miniaturization and high integration as much as possible, wiring materials with low resistance are needed. In MOSLSI's, in particular, poly-Si is widely used as the material for wiring (as word lines or bit lines in memories) as well as for gate electrodes. However, because of the high resistivity of poly-Si at 1 mΩ·cm, signal delays are more conspicuous in MOSLSI's with poly-Si wiring than in MOSLSI's with multilayer Al wiring.

Table 2. Proportional Contractions of MOSFET Elements Under a Fixed Electric Field Condition and Performance

MOSFET parameter	Scaling factor
Element measurements L, W, X_{ox}	1/k*
Density of substrate impurities	k
Power source voltage	1/k
Electric field	1
Electric current	1/k
Gate delay	1/k
Power consumption/element	$1/k^2$
Current density	1
Speed x electric power/element	$1/k^3$

*k > 1

k : Scaling factor measurements
L : MOS transistor channel length
W : MOS transistor channel width
X_{ox}: MOS transistor gate oxide film thickness

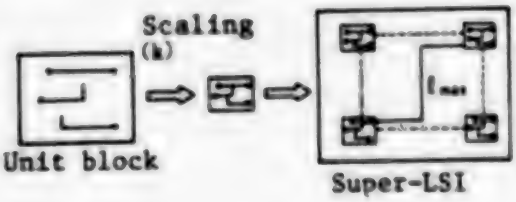


Figure 1. Super-LSI Achieved by Synthesis of Unit Blocks

Table 3. Drops in Characteristics by High-Level Integration of LSI and Wiring Resistance

Unit Block Scaling and Wiring

Wiring parameter	Scaling factor*
Measurements	1/k
Resistance	k
Capacity	1/k
Line capacity	1/k
Signal delay in wiring	1
Voltage drop in wiring	1
Current density in wiring	k

Synthesis of Unit Blocks and Wiring

Wiring parameter		Scaling factor*
Wiring measurements within a unit block		1/k
Long wiring measurements	l_{max}	S
"	resistance	K^2S
"	capacity	S
"	line capacity	S
"	signal delay	K^2S^2
"	voltage drop	kS
"	current density	k

*k, S > 1

S: Long wiring (connections between blocks) scaling factor

3. Properties Required of Low-Resistance Gate Electrodes

A MOSLSI is basically an assemblage of MOS transistors. The gate electrode controls the flow of carriers in the active domain of a MOS transistor, and it is the most important part of an MOSLSI. Thus, the gate electrode must have a number of specific properties, as shown in Table 4. In developing new materials for gates that will substitute for poly-Si, their structures and their processing technology need to be examined in terms of the properties listed in Table 4. In sum, a low-resistance gate must have a resistivity lower than that of poly-Si (phosphor doping) by an order of magnitude or higher, yet must not only be compatible with the existing LSI manufacturing process but have a MOS stability equivalent to poly-Si gates. In terms of material properties, the gate must be able to withstand heat treatment at temperatures as high as 1000°C—the temperature used in the existing Si process. And when the thermal allowance is taken into account, the gate should not melt and should remain stable even at temperatures up to 1200°C. What all this means is that the candidates for low-resistance gate electrodes are inevitably narrowed down to those metals with high melting points or their compounds (silicides). A number of methods for forming thin films out of these metals with high melting points have been developed.

Major techniques among them are the sputtering method, the chemical vapor deposition (CVD) method, and the electron beam vapor deposition method. To form WSi_x thin films for use as WSi_x/poly-Si gate electrodes, which are used in 1M D-RAM's, either the sputtering method or the CVD method is generally used. The silicides with high melting points described in this paper are produced mainly by the sputtering method.

Table 4. Requirements for Gate Electrode and Wiring

Item	Contents
Material	Resistance lower than that of poly-Si by more than one order of magnitude
	Melting point higher than that of Si
	Oxide formation energy higher than that of SiO_2
	High adhesion to SiO_2
	Consistency in thermal expansion equal to that of Si
	Resistance to oxidation
	Resistance to chemicals
	Stable junctions with Al
Impurities	Low alkaline metal impurities
	Low radioactive impurities
MOS characteristics	Low fixed electric charge, low interfacial level
	High hot carrier resistance

4. Polycide Gate Electrodes

A gate electrode with a structure in which polycrystalline Si films doped with impurities such as silicides are stacked up is called a polycide (poly-Si/silicide) gate. The silicide is a layer grown with the objective of lowering the wiring resistance to a level about an order of magnitude lower than that of the poly-Si gate. The poly-Si layer underneath it guarantees stable MOS characteristics on a par with the conventional poly-Si gate. WSi_2, $MOSi_2$, $TaSi_2$ and $TiSi_2$ are generally used for the silicide layer, but in some cases silicide layers of Co or Pt are used. $TiSi_2$ with a resistivity ranging from 15 to 25 $\mu\Omega\cdot cm$ features a wiring resistance that is lower by one-third to one-fifth than WSi_2 (45 to 75 $\mu\Omega\cdot cm$), $MoSi_2$ (80 to 100 $\mu\Omega\cdot cm$) and $TaSi_2$ (40 to 70 $\mu\Omega\cdot cm$) (Figure 2). But in the case of the $TiSi_2$/poly-Si structure, there sometimes occurs a phenomenon in which, while it is being subjected to high temperature heat treating for LSI production, the Ti diffuses inside the poly-Si to reduce the gate SiO_2, and this causes a drop in its dielectric strength. This is because changes in the standard free energy, caused by the reaction between Ti and SiO_2, are negative (Figure 3). In the case of W and Mo, the energy changes brought about by the same reaction are positive, so their reaction with SiO_2 during the

manufacturing process does not lead to a drop in the yield of LSI's. For these reasons, the $MoSi_2$/poly-Si gate electrode and wiring structure have been chosen for the manufacture of 256K D-RAM's (and some 64K D-RAM's) in Japan.

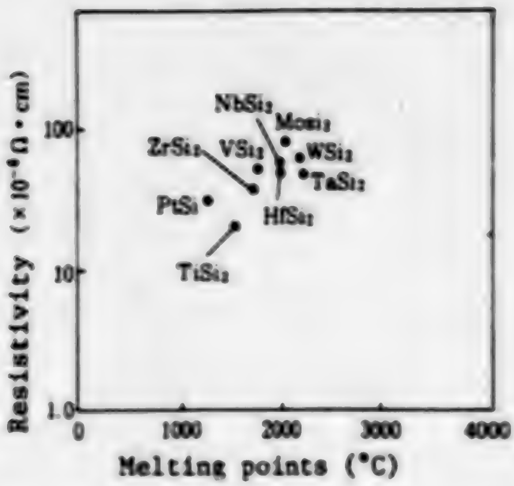


Figure 2. Resistivities (films) and Melting Points of Transition Metals, Silicides

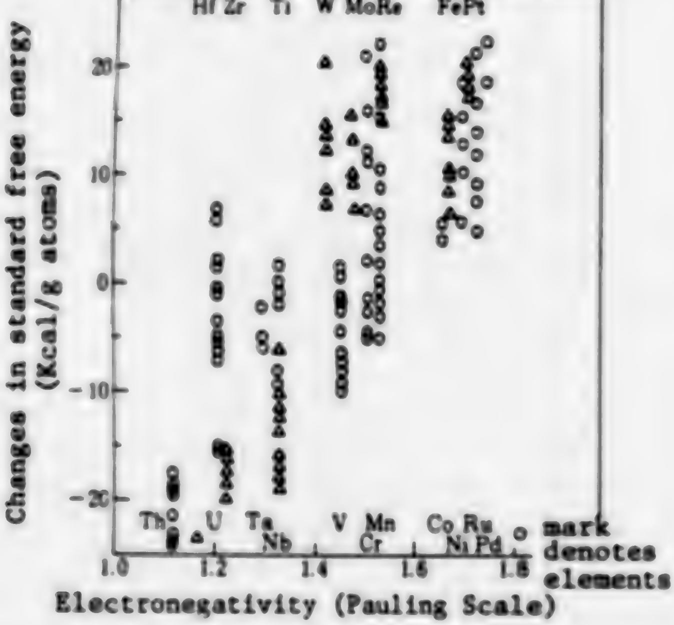


Figure 3. Relationship Between Changes in Standard Free Energy Accompanying Reaction Between Metal and SiO_2 and Electronegativity

Thanks to an epochal growth in CVD technology, WSi_2 has come to be employed in the manufacture of 1M D-RAM's and subsequent generations of devices. In the United States, the majority of semiconductor makers are employing $TiSi_2$/poly-Si gates because of their low resistance features.

In the foregoing the author has briefly described the polycide gate electrode. The following presents the results of studies I have conducted on the role of Si in the $MoSi_2$/poly-Si gate electrode.

I shall first take up the Si layer. To make clear the effect of the poly-Si layer, I will present the results of a stability comparison of MOS characteristics between a MOS capacitor with a Mo metallic gate having no poly-Si layer and a MOS capacitor with a "Mo/poly-Si" gate electrode--an electrode in which a poly-Si layer is sandwiched between its Mo and SiO_2 layers. In the Mo gate, where the Mo layer comes into direct contact with the SiO_2, alkaline metal impurities contained in the Mo film diffuse through the SiO_2 during the high-temperature heat treatment process used in the manufacture of MOS capacitors. These impurities alter the flat band voltage V_{FB}- of the MOS device in operation (MOS transistor's response to its threshold voltage). Figure 4 shows the relationship between the nitrogen annealing time at 1000°C for the manufacture of a MOS capacitor and changes in the flat band voltage in the vicinity of an acceleration life test (BT-test*). As Figure 4 shows, inserting a poly-Si layer between the Mo layer and the SiO_2 greatly improved V_{FB} fluctuations immediately before and after the test.

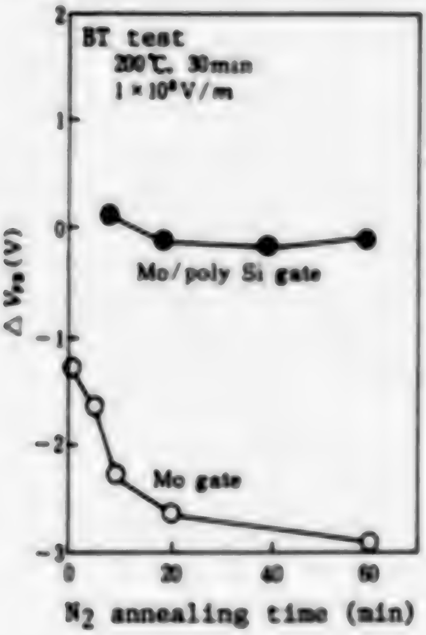


Figure 4. Gate Electrode Structure and MOS Stability (annealing temperature: 1000°C)

*In this test, a sample heated to about 200°C was subjected to a bias voltage so that the movable ions from the alkaline metals contained in the SiO_2 will accumulate on the SiO_2/substrate interface. The number of movable ions is measured by observing changes in the flat band voltage before and after the test. This technique is used for stability evaluation of the MOS device.

The effect of poly-Si in stabilizing the MOS characteristics can also be seen in the case of the interface level.

Figure 5 shows the relationship between gate electrode structure and interface level. It shows that in the Mo/poly-Si gate, a low interface level equal to that of a poly-Si gate can be obtained. Next, I shall describe the role of silicon inside molybdenum silicide.

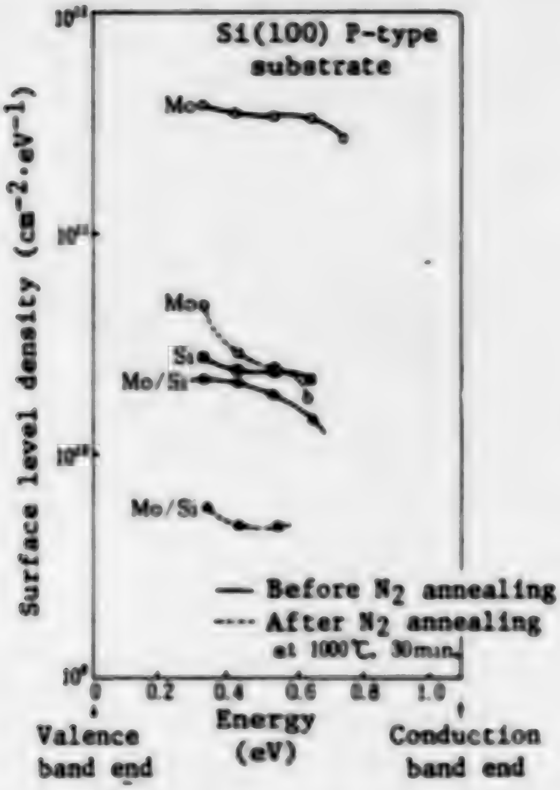


Figure 5. Comparing Surface Level Densities for Various Gate Electrodes

Figure 6 shows the relationship between changes in the internal stress of a silicide film while it is being heat treated and the amount of Si. When Si is not present in the Mo layer, heat treatment leads to a reaction between the Mo and Si, producing a silicide. The reaction-induced formation of $MoSi_2$ results in a volume reduction of about 25 percent below the pre-reaction level. The large change in stress immediately before and after the heat treatment leads to defoliation of the silicide film. Against this, when Si is impregnated in advance in the Mo layer, the consumption of Si from the poly-Si layer, which accompanies the reaction, is constrained, and defoliation caused by the formation of silicide is mitigated. The amount of Si that needs to be impregnated beforehand is more than the stoichiometric composition X = 2.0 (in mass percent, 37 weight percent) of $MoSi_x$—the most stable layer of the two-dimensional alloy systems of Mo and Si. Increasing the amount of Si too much leads to increased resistance on the silicide layer, so the stoichiometric composition is generally set at X = 2.5 ∿ 3.0.

Si inside a silicide film has the effect of preventing the silicide from peeling thanks to the effect of suppressing reaction between the poly-Si and the Mo. Furthermore, the Si prevents the oxidation of Mo while being subjected to a high-temperature heat treatment and also prevents the deposition of oxygen impurities trapped inside the film while it was being grown on the poly-Si interface. One feature of silicides is that they can withstand the effects of chemicals much better than their component metals, such as Mo, alone. Because of their excellent features, they came to be used earlier than other materials in the metallic gate electrodes described below.

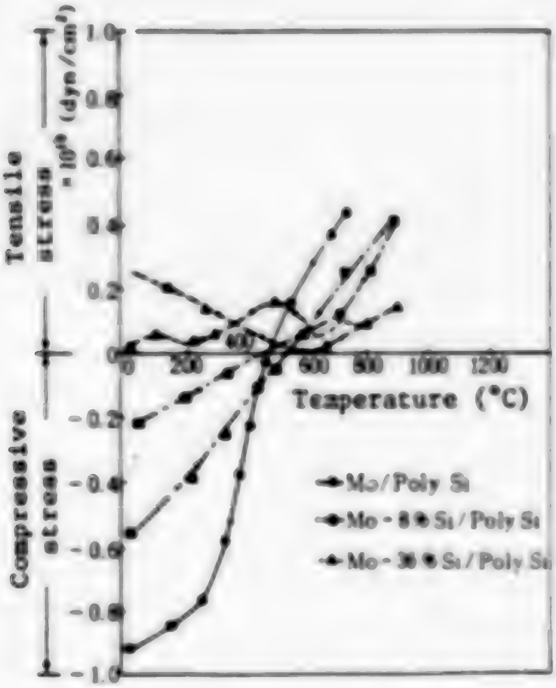


Figure 6. Si Density and Stress Inside Film

5. Gate Electrodes of Metals With High Melting Points

The research on metal gates with high melting points started earlier than the study of polycide gates. The publication of the first report on the metal gates coincided with the initial period of research on poly-Si gates. Mo and W were studied as electrode materials. These metal gate electrodes, however, proved unsatisfactory for commercial application because of their defects:

1. They were easily oxidized and evaporated in the course of the high-temperature heat treatment used in manufacturing semiconductors; and

2. As shown in Figure 4, alkaline metal impurities such as dodium easily become incorporated into their metal films, which creates their unstable MOS characteristics.

Metal gate electrodes, however, once again attracted attention with the appearance of megabit LSI's. In fact, studies of Mo and W are under way in an effort to find a way to use them as low-resistance gate electrodes for the ultra-LSI expected to come after the 64M D-RAM. Oikawa et al. have been engaged in research on Mo, while Yamamoto et al. have been conducting research on W. The following discussion focuses primarily on W gates.

W and Mo are used as gate electrodes for the following reasons:

1. These metals make it possible to obtain low resistivity films ranging from 6 to 12 μΩ·cm; and

2. As described above, the oxide-forming energies of metals are higher than Si oxide-forming energies, and hence these metals undergo little reaction with SiO_2.

These metals are formed by the sputtering method using Ar gas. In this process, the internal stress of the metal film can be freely changed by controlling the pressure of the Ar gas (Figure 7). The conditions governing the Ar gas pressure, i.e., the film's internal stress, are determined by taking account the need to ensure that the film will not peel off when subjected to high-temperature heat treating and that its MOS characteristics will be stable. For information on the relationship between the internal stress of a gate electrode and the resistance to the pressure of the gate insulating film or the hot carrier resistance of a micro-MOS transistor, the reader is referred to the author's earlier writings.

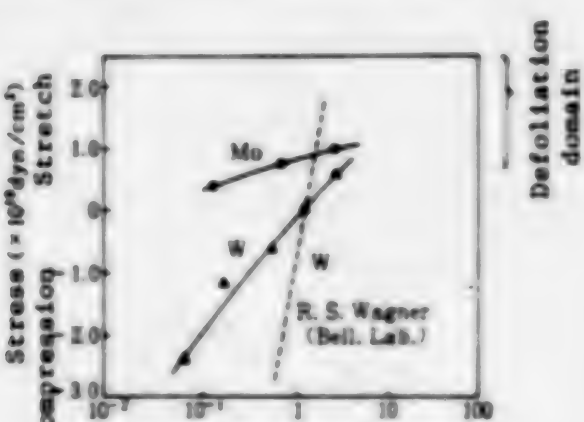


Figure 7. Dependence of Internal Stress on Sputtering Ar Pressure

The biggest impediment to the practical use of metal gate electrodes was that there did not exist a technology for growing thin films relatively free of alkaline metal impurities.

The author and others have studied possible ways to raise the purity levels of metal targets for sputtering--the source of alkaline metal impurities

inside a film. Figure 8 shows the manufacturing process of a high-purity sputtering target. The key in this process is the achievement of a high level of purity in the target that makes it possible to use an electron beam dissolution process. The level of sodium impurities contained in a thin W film, fabricated by using the target, is below 10 ppb, a figure about three orders of magnitude smaller than when conventional targets are used.

High-purity W powder
↓
Compacting and sintering
↓
Electron beam dissolution
↓
Ingot
↓
Compression forging
↓
Mechanical machining
↓
Etching
↓
[High-purity target]

Figure 8. Manufacturing Process of High-Purity W Target

Figure 9 shows the relationship between the amount of impurities contained in a target and the degree of contamination by movable ions. These data were obtained by measuring the electric characteristics of a MOS capacitor manufactured using the target. When using high-purity targets, the degree of contamination is at a level where the contamination can barely be detected or cannot be detected at all. For this reason, the application of W gates to LSI's would pose no problem in terms of characteristic changes created by contamination from movable ions.

Another factor impeding the practical use of gate electrodes made from metals with high melting points was that these metals become oxided and evaporate during the course of heat treatment even in a nonoxidation atmosphere such as nitrogen or argon if any oxygen is present on the order of several ppm--an important defect. In the silicon LSI process, however, in order to obtain high-reliability MOS transistors it is essential to remove the SiO_2 film formed in the vicinity of the gate electrode on the Si substrate surface, which is contaminated in the course of dry etching the gate electrode and in the process of ion implantation for the fabrication of the source/drain, and then to reoxidize the surface. If the W or Mo gate process is to be compatible with the Si process, it is necessary that there be a system with an environment in which these metals can coexist with Si, and where only Si is selectively oxidized. Previously, this was thought to be impossible. Based on theoretical and experimental studies, the author and others have developed a new heat treatment method that will make possible

the selective oxidation of Si--considered impossible until now--by drawing on the following thermodynamic considerations.

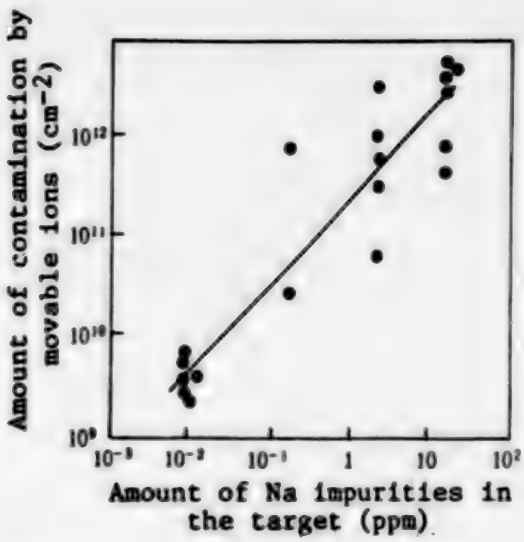


Figure 9. Amounts of Na Impurities in Targets and Contamination by Movable Ions

The reduction reaction of tungsten oxide by hydrogen, or the oxidation reaction of tungsten by water, is given by the formula:

$$WO_3 + 3H_2 \rightleftarrows W + 3H_2O \quad (1)$$

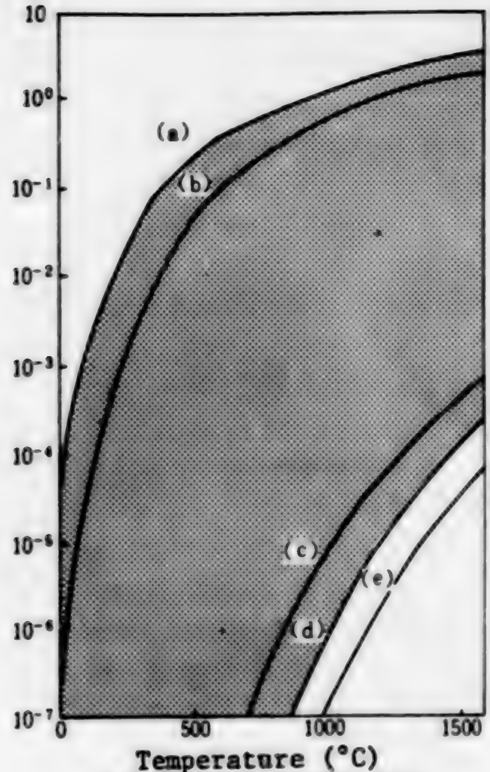
The variations in Gibbs free energy following the progression of the reaction in the right direction, ΔG, is expressed as

$$\Delta G = 24800 + 10.2 \log T - 52.4\,T \quad \text{(cal/gatom)} \quad (2)$$

where T is the absolute temperature. Since a relationship exists between ΔG and the vapor pressures P_{H2} and P_{H2O} of H_2 and H_2O at the time of thermal equilibrium, expressed by the following equation

$$\Delta G = -4.574\,T \log\,(P_{H2O}/P_{H2})^3 \quad (3)$$

from formulas (2) and (3), the equilibrium vapor pressure ratio between H_2 and H_2O, $k = P_{H2O}/P_{H2}$ can be calculated for any temperature. Figure 10 shows the results of the calculations for Si, Mo, Ta and Ti, in addition to W.



(a) $W + 3H_2O \rightleftharpoons WO_3 + 3H_2$

(b) $Mo + 2H_2O \rightleftharpoons MoO_2 + 2H_2$

(c) $2Ta + 5H_2O \rightleftharpoons Ta_2O_5 + 5H_2$

(d) $Si + 2H_2O \rightleftharpoons SiO_2 + 2H_2$

(e) $Ti + 2H_2O \rightleftharpoons TiO_2 + 2H_2$

Figure 10. Dependence of Equilibrium Vapor Pressure Ratio $P_{H2O}P_{H2}$ on Temperature

The figure indicates that in the case of tungsten, for example, the value of k is about 1 (one) at 1000°C. Therefore, formula (1) proceeds in the right direction, i.e., the reduction of WO_3 when $k < 1$, whereas it proceeds in the left direction, i.e., the oxidation of W, when $k > 1$. Such an argument is applicable to Si as well. In this case, the value is $k \approx 10^{-6}$ at 1000°C. From the foregoing arguments, one can see that carrying out a heat treatment in the moisture-added hydrogen atmosphere within the region defined by the equilibrium vapor pressure curves for W and Si in Figure 10 will make it possible to selectively oxidize Si even in the presence of W. Heat treatment in this region is called the WH (wet hydrogen) treatment method. As can be seen from the figure, this heat treatment method is applicable to Mo or Ta but is not available for Ti. The newly developed WH heat treatment method was employed in the manufacture of W-gate MOS transistors. The results confirm that the MOS characteristics of these products are drastically improved compared to products that have not been treated with the new heat treatment technique:

1. The pressure resistance of the gate insulating film has been improved by about 2 to 3 V; and

2. Contamination by impurities that find their way in during the manufacturing process, such as movable ions, can be reduced.

The improved MOS transistor is not inferior to conventional transistors with poly-Si gates in terms of transistor characteristics.

6. Conclusion

In this paper the author has focused on the application of metals with high melting points in gate electrodes and their various applications to LSI's. Polycide gate electrodes have already been put to practical use, and we are currently progressing from the era of Mo silicide to the era of tungsten silicide. In particular, silicides are becoming indispensable as materials for the manufacture of megabit LSI's. The reason why polycide gate electrodes overtook gates made of metals with high melting points in terms of practical application--although research on the latter started earlier--can be explained by the fact that the former have an edge over the latter in terms of the manufacturing process and MOS characteristics. We consider, however, that thanks to the development of the various technologies for improving their reliability described above, gates made of metals with high melting points have fully reached the level of practical application. Therefore, we feel that semiconductor makers who overcome their ingrained conception that "high-melting-point metal gates have low reliability" will become the pioneers in the practical application of ultra-LSI's. In addition to their use as gate electrodes, metals with high melting points are beginning to find applications in a wide range of fields, such as multilayer wiring, planation of microcontact holes (tungsten selection CVD technology) and barrier metals in sections where Al wiring and Si substrates come into contact. Thus they will be important materials for future LSI's.

References

1. R.H. Dennard et al., IEEE J. Solid-State Circuits, SC-9, 259 (1974).

2. B.L. Crowder and S. Zirinsky, IEEE Trans. Electron Devices, ED-26, 369 (1979).

3. R. Pretorius et al., Solid-State Electronics, 21, 667 (1978).

4. Yamamoto et al., Material by the Research Society on Semiconductors and Transistors, Institute of Electronics and Communication Engineers of Japan, SSD 82-98 (1982).

5. D.M. Brown et al., Solid-State Electronics, 11, 1105 (1968).

6. L.L. Vadasz et al., IEEE Spectrum, 28, October (1969).

7. Oikawa and Amazawa, OYO BUTSURI [APPLIED PHYSICS], 47, 192 (1978).

8. Oikawa and Amazawa, A Collection of Draft Papers for Presentation at the 42nd Meeting of the Japan Society of Applied Physics (Spring), 714 (1981).

9. N. Yamamoto et al., Ext. Abs. 15th Conf. Solid State Devices and Materials, 217, A-6-1 (1983).

10. N. Yamamoto et al., J. of Electrochem. Soc., 133, 401 (1986).

11. N. Yamamoto et al., IEEE Trans. Electron Devices, ED-34, 607 (1987).

12. N. Yamamoto, Ext. Abs. 19th Conf. Solid State Devices and Materials, 415 (1987).

20114/6091

Functionally Gradient Materials Workshop No 1

Table of Contents

43067596 Tokyo FGM NEWS in Japanese May 88 p 27

[Text]

Expectations for FGM

43067596 Tokyo FGM NEWS in Japanese May 88 pp 1-3

[Article by senior research member Yoshikuma Ishikawa, Mitsubishi General Research Laboratory on expectations harbored for functionally gradient materials (FGM)]

[Text] There has never been a time when the development of new materials has been desired as much as today, and the challenge represented by this demand has been strongly taken up by the whole nation. As is known, the appearance last spring of types which do not require rare earth elements (Bi-Sr-Ca-Cu oxide system), which should be called the Hinomaru (Rising sun) superconductor, causing an international sensation following the lull in superconductor fever, has led to a renewed acceleration in the development of new materials since the beginning of the new year. At the same time, the FGM's resulting from Japan's original development have finally come to be recognized, and the fact that a joint industrial and governmental materials research society on superconducting materials has been established is an epoch-making fact worthy of special mention in the materials development of Japan.

According to an analysis based on the fluctuation view of business cycles, the budding of technological renovation supporting the next economic growth will become concentrated around the beginning of the 1990's, and these new materials and new raw materials are indeed an appropriate means to trigger the next fluctuation. In the case of FGM, nothing indeed is more gratifying than the fact that a good start has been made possible by the great expansion of the small workshop program, the large increase in the number of participating enterprises, and the successive inquiries coming from abroad. Although the reason why these materials are attracting such attention is due in large part to the zeal of all parties concerned, including the National Aerospace Laboratory which is the development center, another reason for this interest is by no means unrelated to the fact that these materials suggest a direction for the future development of new materials.

FGM's are considered the first of the fusion materials in the material technology trend ranging from the conventional "separation type" centered around separate functions, via the "composite type" aiming at the synergism of dominant property among component elements, up to the "fusion type" in which the scope reaches the microscopic level whereby even the distinction between different types of materials is not clear.

Since intended gradient functions can be selected in these materials by means of multiple changes in the composition and structure in regard to thickness and a variety of materials can be selected for combination, new functions totally different from those now common are available. When one reflects that this type of heterogeneous material was often conventionally treated as a defective material, it appears that we are living in a quite different age.

Although opinions are divided on the background of why such a new materials concept has appeared, it appears that after all it is largely due to the inception of such huge projects as the future aerospace and energy projects, the development of materials by computer-aided means, etc.

The future spacecraft regarded as the immediate objective is a gigantic project in which various advanced nations such as the United States, the EC nations, and Japan are engaged in a fierce development competition with each other, with the development target set at the beginning of the 21st century. The breakthrough in this gigantic project, after all, solely depends upon whether or not the advanced technologies in regard to materials have reached the appropriate level at the present stage. For example, the achievement of the appropriate temperature for each element of the space shuttle based upon the estimated speed of Mach 8 (eight times the speed of sound) when it reenters the atmosphere is as shown in Figure 1. The temperature range reaching the highest level as a result of aerodynamic heating is at the nose and at the inlet of the propulsion system, where it reaches a temperature of almost 1,800°C. Moreover, the shuttle is also exposed to a temperature as low as -253°C when the liquid hydrogen engine is used as the propulsion system and it becomes an incredible "fireball flying shuttle with a built-in ultralow-temperature tank."

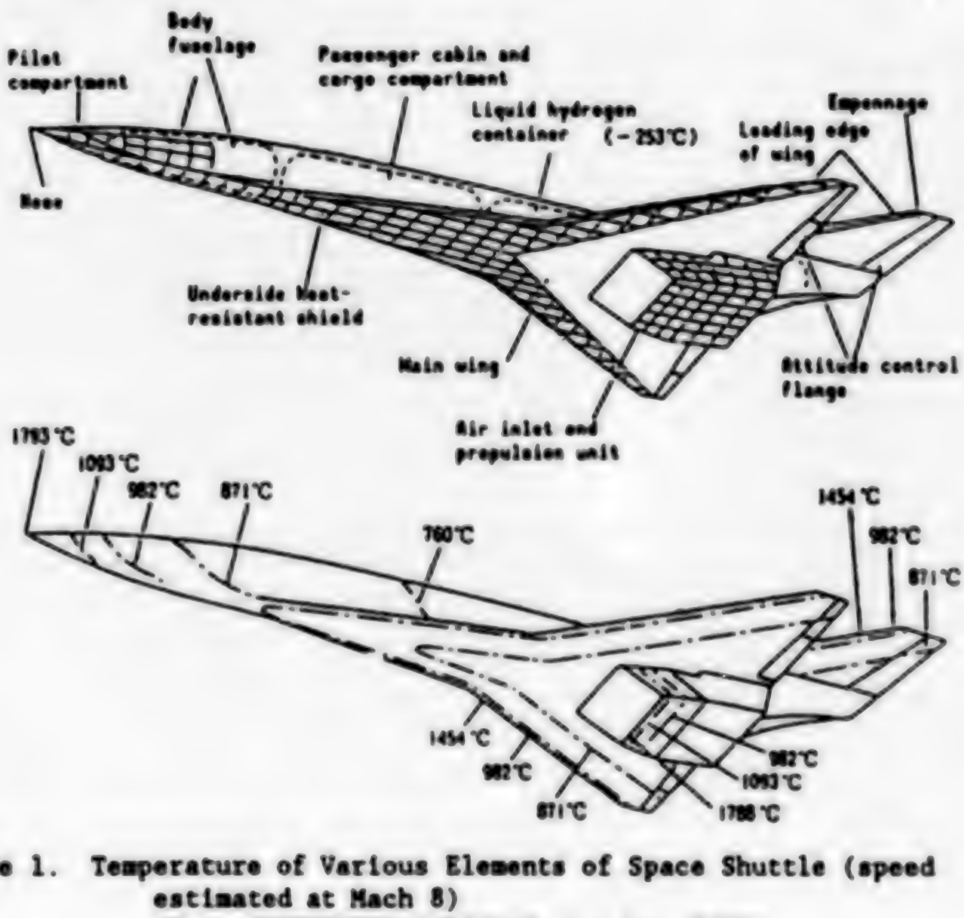


Figure 1. Temperature of Various Elements of Space Shuttle (speed estimated at Mach 8)
(From SCIENTIFIC AMERICAN, October 1986)

Among the various materials presently considered as candidates for fuselage materials for such spacecraft, the use of various inorganic system composite materials for high-temperature parts is under consideration by the United States and other countries, and a competition in this, including FGM's, can be expected in the future. It is evident that the key to the success of a space shuttle operating in a harsh environment lies in the propulsion system, and the material's technology, the importance of the material for this system, is also great. After all, materials have reached the position of the latest stage high technology. We recently came across an expression to this effect also in the United States.

Up to now, our discussion has centered on heat-resistant characteristics for the aerospace field, which is the immediate area of concern. However, the concept of functionally gradient materials is not limited to such thermal and mechanical functions. It is believed that gradient materials will be oriented to multiple functions in the future, and that the range of application will also be diversified, as indicated in Table 1. Expectations are especially high in regard to the polymer and biological areas, in which the practical use impact will be great.

Table 1. Examples of Functions and Applications of Functionally Gradient Materials

Anticipated gradient function	Application example
Mechanical and thermal functions	Thermal stress relaxation type heat-resistant material
Electrical and magnetic functions	Mount integrated type sensor
Chemical function	Functional polymer film
Optical function	Refractive index distribution optical fiber
Nuclear function	Low Z component gradient type furnace wall material
Biological function	Porosity distribution artificial dental root

(Prepared by MRI from National Aerospace Laboratory catalog)

For FGM's to become widely accepted as industrial materials in the society, taking into account the environment and characteristics required, the issues and problem points of these materials must be ascertained and appropriate countermeasures taken.

Therefore, I wish to narrow down the development of elements of new materials to match the needs and seeds; to list the three elements of materials and other development; and especially, to make clear the expectations harbored for materials in their development.

1. Matching Needs and Seeds

The matching of needs and seeds is important for the new materials to be accepted for use as industrial materials, and a needs orientation is also required during research and development.

2. Three Elements of Materials Development (Design, Synthesis, Evaluation)

The new materials must be endowed with superior characteristics not found in existing materials, and thus the focus is on development of the technology to stack up the materials components while maintaining control at the atomic and molecular levels. Also, the elaboration of a materials theory as a basis, the consolidation of a database in regard to both synthesis and evaluation, and the utilization of artificial intelligence must not be neglected.

It is necessary to establish a process whereby the synthesis conditions are computed in order to obtain the optimum gradient function and the components are continuously changed by freely using various synthesis technologies. Complicated, fine processes that are not generally used with existing materials are also necessary, but since they are apt to incur high cost, special attention must be given to this aspect as well as to the reworkability of the materials.

Testing and evaluation methods must be standardized when the materials are put to practical use, and their reliability as materials must be guaranteed. When the materials are upgraded evaluation becomes difficult and conventional methods cannot be used, so the evaluation method itself becomes the object of development. The three elements of materials development are strongly interrelated, so the three elements of development must proceed in close concert.

3. Other (Environment, Recycling, Cost)

The period of acceptance as long as the result was a material excelling in function has ended; now, a prerequisite of the new materials is that the environment not be damaged. The characteristic as a fusion material is a factor to limit ways of dumping and recycling the material. Since at present there are many kinds of new materials that entail minimum production and they are lacking in marketability, consideration must be given to cost-effectiveness. Despite the great expectations harbored for new materials development, it appears that the issues involved are inexhaustible.

While it is often said that "the one who controls the material also controls the technology," there are also many cases of technological development that came to a halt because of the lack of a promising material.

We cannot forget the historical fact that at the beginning of the century, the development of a then revolutionary aircraft which did not require a propeller was stalled for a long time due to the delay in the development of materials for a jet engine. Now, almost a century later,

superheat-resistant materials oriented for use in future aircraft are demanded, and this time I look forward by all means to the timely support of an appropriate material. The course of systematic research and development of functionally gradient materials has just started, and the journey will be a long one. I look forward with all hope that a steady, bold effort will be made, centered on the New Materials Workshop, to meet the challenge to put the new materials to practical use.

Discussion With NASA's Dr DiCarlo

43067596 Tokyo FGM NEWS in Japanese May 88 pp 4-5

[Report on discussion on the FGM Workshop during the visit to the Sumida branch of the National Aerospace Laboratory by NASA's Dr James DiCarlo]

[Excerpt] Dr James A. DiCarlo of the Lewis Research Center of NASA visited the Sumida branch of the NAL on 29 March. Seventeen members of the FGM Workshop group met with him, and seven of the representative members described the work carried out by the FGM Workshop. Then Dr DiCarlo gave a lecture on the research into heat-resistant composite materials at NASA, and held a discussion with the Japanese research members.

Presentation Made on FGM and Discussion With Dr DiCarlo

First of all, an outline was given of FGM developments by the FGM subcommittees. The presentation included an explanation of the overall plan (Niino, the NAL), materials design (Hirano, Daikin Industries, Ltd.), structure control (Kawasaki, Tohoku University; and Sata, Government Industrial Research Institute, Tohoku) and characteristic evaluation (Shoji, Tohoku University; Nagata, Mitsubishi Heavy Industries, Ltd.; and Kumakawa, NAL). After this explanation, the following exchange of opinions ensued between representative of the FGM Workshop and Dr DiCarlo of NASA.

FGM: What is your first impression?

NASA: It was very impressive. The approaches made on the issues were good. Challenges have been made from various aspects, with universities, research laboratories, and civilian enterprises acting in concert, and I believe that this has been organized extremely well on the whole. By the way, how often do the members of the FGM gather? Isn't it a strain physically?

FGM: Representatives of the 17 main organizations gather quite frequently. Since Japan is a small country, by using the train they can gather in a little more than 2 hours.

NASA: It is working out well at present. In regard to the divulgence of classified information among enterprises, this can be solved as a managerial problem.

NASA: Are the applications limited already? In regard to aerospace, I do not believe that all of the metal-ceramic combinations in the presentation made today can be used.

FGM: We are considering aerospace-related nonstructural materials and, for example, engine parts that involve forced cooling. However, the development of various functionally gradient materials is our main target, and we wish to consider a wide range of applications.

NASA: If you are considering aerospace use, oxidation-resistance may also become a major topic. What fields do the people in this project represent? I have the impression that the roles of the oxidation mechanism and the reaction of metals-ceramics have been left out.

FGM: There are also other research members who are engaged in important topics besides those gathered here today.

NASA: How do you view the subject of fineness, or the presence of porosity?

FGM: Porosity is one means for stress relaxation.

NASA: What means do you use for evaluating the ability of materials to withstand a difference in temperature of 1,000°C?

FGM: It is now being installed at the NAL. (After this, there were questions and answers on the details and quality of the equipment being installed.)

NASA: I did not know anything about FGM until today. I will introduce the subject of FGM to NASA, but I wish that you would also spread the word abroad. I think you should write a paper on it in English.

FGM: It is still only 6 months since our inauguration, and we intend to proceed steadily with public relations activities from now on.

NASA: The same topics in regard to aerospace are being pursued by NASA. I wish you success in this project, and I am sure that you will be successful.

Outline of Workshop

43067596 Tokyo FGM NEWS in Japanese May 88 p 6

[Report by Yasuo Kubota, National Aerospace Laboratory, outlining the FGM Workshop research topics]

[Text] The workshop offers a place for informative lectures on specialized topics, a place for questions and answers, with the various research members engaged in FGM research invited to attend as lecturers.

Such lectures are held four times a year, the lecture participants as a rule are limited to the workshop members (two persons per organization) and the

objective is to provide lectures that serve the research and development purposes of each member present by enabling intimate conversation with the lecturers. It differs fundamentally from symposiums, in which the results obtained are briefly and widely announced to the public.

Table 1. Research on Basic Technology of FGM Development for Thermal Stress Relaxation

Research Topics of Structure Control Subcommittee and Research Organization in Charge

Major topic	Subtopic	Research objective	Research organization in charge
I. Research on composition control technology by physical and chemical vapor deposition method	① Physical vapor deposition method	Establishment of composition control conditions of coating layers by the PVD method	National Research Institute for Metals, Agency of Industrial Science and Technology
	② Chemical vapor deposition method	Establishment of functionally gradient material synthesis technology of SiC/C system by the CVD method	Metallic Materials Laboratory, Tohoku University
	3 Physical and chemical vapor deposition method	Extraction of effects and problem points anticipated in the physical and chemical vapor deposition methods	Sumitomo Electric Industries, Ltd.
II. Research on structure control technology by particle method	① Particle injection method	Conducting research on injection filling, compaction sintering fining behavior and fine structure control of ceramic powder, heat-resistant metallic powder and ceramic whiskers. Ascertaining the thermal and mechanical characteristics of nongradient sintered materials	Materials Processing Course, Faculty of Technology, Tohoku University

[continued]

[Continuation of Table 1]

Major topic	Subtopic	Research objective	Research organization in charge
	② Research on laminate array technology by thin filming of particles	Development of wide area array and molding sintering technology by the laminate method for the preparation of large-sized functionally gradient members consisting of ceramic material and Ni group superalloy	Nippon Kokan K.K.
III. Research on laminate molding technology by thermal spray method	① Independent thermal spray technology of different types of particles	Studying conditions to develop gradient composition laminates consisting of metal and ceramics by using two thermal spray units	National Research Institute for Metals, Agency of Industrial Science and Technology
	2 Simultaneous thermal spray technology of different types of particles	Establishment of gradient thermal spray laminate structure control technology	Nippon Steel Corp.
IV. Research on structure control technology by self-exothermic reaction method	① Reaction control technology	Promoting the complexing of boride and metallic system structures and establishment of the control technology of reaction, composition and fine structures	Industrial Science Laboratory, Osaka University
	2 Wide area control technology	Establishment of so-called wide area synthesis technology whereby a large-sized compact is obtained without the use of high pressure. Trial manufacture of FGM's of the boride-metallic system based on this technology	Government Industrial Research, Institute, Tohoku

Note: Items marked O begun in 1987; Items not marked begun in 1988.

Since the first allocation for FGM research aiming at the development of superheat-resistant materials appeared in the 1987 national budget under the science and technology promotion adjustment expenditure category, inevitably the number of lectures related to this research will tend to increase this year. Furthermore, we plan gradually to include lectures on research that is conducted independently along those on research under the promotion adjustment expenditure, as well as research in various fields other than that of heat-resistant materials. I wish to solicit the cooperation of all workshop members.

Since this is the first workshop assembled, it is being held under the theme of "how to control gradient composition" and it is centered on introducing this research and on providing lectures on the structure control method.

Materials Design Subcommittee

43067596 Tokyo FGM NEWS in Japanese May 88 p 7

[Report by Kenji Wakashima, Precision Engineering Laboratory, Tokyo Institute of Technology, introducing the work of the Materials Design Subcommittee]

[Text] Functionally gradient materials (FGM) are comprised of various types of materials, including those of the ceramics-metals system. They are composite materials in a broad sense, but they differ from ordinary composite materials in that the microstructure has a gradient form that is microscopically changed. The focus of "materials design' in such a heterogeneous material lies in optimizing the microstructure and the macrostructure of the material in order to achieve a manifest function in accordance with the purpose. As is known, the materials design technology for FGM must ultimately be established in accordance with the individual purpose of use, as the functions required for materials vary widely. However, no few problems remain to be studied at the basic level in order to achieve this goal. Next, I will introduce in particular the activity of the Materials Design Subcommittee on the "research on the basic technology of FGM development for thermal stress relaxation" conducted under the science and technology promotion adjustment expenditure category.

This research is aimed at dealing with the basic problems encountered in controlling the FGM process--that is, realizing the macroscopic gradient of the microstructure of thermal stress fracture, which has especially become a problem in the ceramics-metals system, together with developing superheat-resistant materials excelling in thermal insulation property as its application. The Materials Design Subcommittee is responsible for the following research themes.

(1) "Research on physical property estimation technology for FGM design"
(2) "Research on an FGM theoretical model and a thermal stress analysis model"
(3) "Research on a computer-aided materials design support system"

In the method established in the research conducted under item (1) (Precision Engineering Laboratory of Tokyo Institute of Technology in charge) the goal was to theoretically estimate the thermoelastic physical property values (elastic constant, coefficient of thermal expansion, heat conductivity, specific heat, etc.), then to consider the thermoelastic physical property values of the constituents and microstructure factors (shape of disperse phase, orientation property, volume percentage, etc.) of mixed materials as given information, and finally to consolidate the input data in a retrieval environment appropriate for thermal stress analysis of FGM. Specifically, we developed a thermoelastic composition theory of polyphase mixed materials based on a microscopic mechanical model which handled the disperse phase as an ellipsoid, and together with formulating a computation method using an elastic constant and coefficient of thermal expansion, we prepared computation programs by means of FORTRAN. In the future, we will also develop similar computation projects on heat conductivity and specific heat. Moreover, we expect to study in detail the estimation accuracy of the physical property values by comparing them with actually measured data.

Under research item (2), (National Aerospace Laboratory of Science and Technology agency in charge), a study will be on a modeling technique for carrying out heat conduction/thermal stress analysis of FGM, and numerical analysis software will be developed based on this. For the time being, on the basis of the hypothesis of the one-dimensional gradient on an infinite plate and infinite cylinder in the thickness direction, thermal stress analysis will be performed when the prescribed temperature difference has been provided on both front and back surfaces. While analysis of thermoelastic stress in the thermal stationary state has been possible up to now, we also wish to conduct an elastoplastic analysis taking into consideration the heat history, and to establish a prestrain design on this basis. Moreover, in the future we wish to conduct analysis of elastoplastic thermal stress in the thermal nonstationary state.

Under research item (3), (Daikin Industries, Ltd., in charge), the target is to develop an expert FGM design system centered on estimating the maximum gradient structure. Although such estimation is an "inverse problem" type, optimization is achieved for the time being by repeatedly executing "regular problem" analysis according to the method mentioned above while changing the initial conditions (parameters that prescribe the gradient distribution). A plastic model material of the fluororesin-silver system was trial manufactured in order to confirm the estimated results by experiment, and the estimated accuracy has been studied by comparing the estimated results with the temperature distribution results measured in the stationary state. Optimization, including sensitivity analysis, will be studied in the future. However, since the realization of artificial intelligence (AI), which has made production knowhow in various structure control technologies the basis of knowledge, is also important, basic s 'les aiming at the construction of a global optimization system including this feature will be promoted.

Structure Control Subcommittee

43067596 Tokyo FGM NEWS in Japanese May 88 p 8

[Report by Ichiro Shioda, National Research Institute for Metals, introducing the work of the Structure Control Subcommittee]

[Text] The gradient composition material used in this research was a material based on a totally new concept not found in conventional materials, and concerted research is being promoted among the Design, Structure Control, and Evaluation Subcommittees. The research responsibilities of the Structure Control Subcommittee are shown in the table previously presented.

As is clear from the table, various studies are being promoted. These studies include physical and chemical vacuum deposition methods, wherein studies are conducted on nano-order microscopic structure control; the particle method, wherein structure control of slightly broad dispersion state is achieved by using fine grains; the thermal spraying method, which is considered advantageous when forming gradient composition materials on the surface of large members; and the self-exothermic method, in which direct forming of large members can be expected without using special high-pressure equipment.

These methods, which have the respective characteristics mentioned above, pertain to the materials system in which we are strong. Studies are being conducted on the Ti-TiN, Cr-CrN, and Ti-TiC systems in the physical and chemical vacuum deposition methods; on the Al_2O_3 and SUS, ZrO_2 and SUS, Ni alloy and Ni-Cry alloy systems in the particle method and the thermal spraying method; and on the TiB_2-Cu system in the self-exothermic method. In 1987, studies were conducted on the formation conditions and characteristics of nongradient materials of various compositions, from metal to ceramics, as the basis for developing functionally gradient materials. Moreover, a preliminary study on gradient composition materials on the basis of the results.

At this first workshop, an outline has been given of the present status of research in order to promote understanding of the concept of FGM.

Characteristics Evaluation Subcommittee

43067596 Tokyo FGM NEWS in Japanese May 88 pp 8-9

[Article by Masayuki Niino, National Aerospace Laboratory, introducing the work of the Characteristics Evaluation Subcommittee]

[Text] The research topics and aims that are presently being promoted under the science and technology promotion adjustment expenditure category are as follows:

(1) Research Technology for Quantitative Evaluation of Local Thermal Stress (Ship Research Institute)

The aim is to develop a technology for evaluating the local thermal stress distribution of functionally gradient materials (FGM) that receive a temperature load by using the laser ultrasonic method and the CT (computerized tomography) method.

(2) Research on Thermal Insulation Performance Evaluation Technology (National Aerospace Laboratory, Faculty of Technology, Shizuoka University)

A high-temperature head basic evaluation test is conducted at an ideal heating site, and thermal insulation and durability tests are conducted at an aerodynamic heating site, a high-temperature gas flow site and a high-speed rotation site.

(3) Research on Thermal Fatigue Evaluation Technology (Mitsubishi Heavy Industries, Ltd.)

The aim is to obtain thermal fatigue life data by conducting a thermal fatigue test using uniform functional materials and FGM material, to ascertain the micromechanics of thermal fatigue and to establish a rule on thermal fatigue life.

(4) Research on Thermal Shock Evaluation Technology (Faculty of Technology, Tohoku University)

The aim is to establish a thermal shock evaluation test method that can be commonly performed by free use of the laser heating method and the AE method to evaluate the characteristics of superheat-resistant materials having various gradient microstructures.

(5) Research on Mechanical Strength Evaluation Technology (Mechanical Technology Laboratory)

The aim is to establish a fracture strength characteristic evaluation method through elucidation of the microscopic fracture mechanism of FGM's under very high temperatures and through development of damage analysis and monitoring technology.

Since an evaluation section typically begins its research work after receiving the results obtained by design and structure control section, no conspicuous results have been achieved in the initial year of the project. However, tests were made using a common ceramics testpiece (brand name, Macoru) for the purpose of promoting standardization of the evaluation level in the evaluation section. The following are the major results obtained.

·Thermal physical property value evaluation test at the National Aerospace Laboratory-Shizuoka University

Measurements of the Macoru testpiece thermal conductivity were made by means of the nonstationary method, the stationary method and the combustion gas

heating method. The degree of coincidence of the various measuring methods was superior for temperatures from room temperature up to 750 K.

•Thermal fatigue test at Mitsubishi Heavy Industries, Ltd.

A thermal fatigue test was conducted using an infrared image furnace with a cooling device. The strength showed a tendency to improve with the increase of cycles in the thermal cycle test, with an atmospheric temperature of 1,300 K and temperature change of 800 K. It is believed that this was due to a residual stress generated in the interface by the softening of the glass.

•Thermal shock test at Tohoku University

The critical laser output density generated by a thermal shock crack was measured using AE, also and it was found that the critical laser output density was 2.2 W/mm^2 in the Macoru material and 12.0 W/mm^2 in zirconia coating material.

The consolidation of test equipment is also being promoted in other evaluation sections, and it is thought that reports on more concrete results can be presented in the near future when many FGM testpieces appear on the market.

Use of CVD Method

43067596 Tokyo FGM NEWS in Japanese May 88 pp 10-13

[Report by Toshio Hirai, Tohoku University and National Research Institute for Metals, on the creation of gradient composition materials using the CVD method]

[Text] **I. Introduction**

Research and development of gradient composition materials was started with the development of thermal insulation materials for space shuttles as the immediate objective.[1] One of the origins of gradient composition materials is a composite material called a "nanocomposite" which contains the ultrafine disperse phase of the nanometer order. It is said that the nanocomposite may become one of the important new materials in the future.

It has been suggested that the goal should not be limited to obtaining the disperse phase of materials alone, but should include compounding (complexing) the various elements.[2] I will consider this a complex material, and call it a fine composite.[3] Materials in which the functions have been made gradient by continuously changing the concentration of the disperse phase or disperse elements form one side of the material to the other side are functionally gradient materials (FGM).

Various methods are available for the synthesis of nanocomposites, fine composites, and FGM. My research group adopted the chemical vapor deposition (CVD) method using gas as the material for the synthesis.

Therefore, research examples using the CVD method will be introduced in this article.

II. CVD Method

The CVD method consists of decomposing the material gas by heat, light, and electrons, generating a chemical reaction and obtaining a solid material. A representative example of a thermal CVD device is shown in Figure 1.

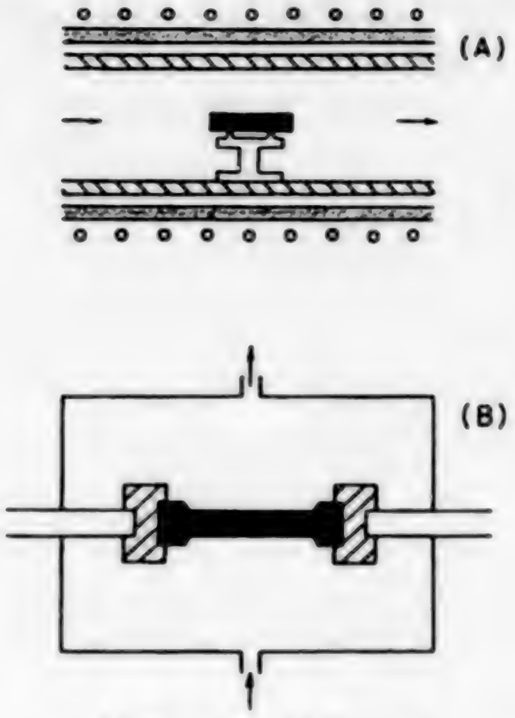


Figure 1. Type of Composite Materials

III. CVD·Nanocomposite

Composite materials synthesized by the CVD method can be largely classified into (I) phase junction type and (II) phase separation (in-situ) type, as shown in Figure 2. In types (I-A and I-B), the target material has been coated on the substrate by the CVD method. In type (I-C), the target material has been infiltration deposited in the gaps of porous fabric cloth made of woven fibers (called CVI). The substrate and CVD material have been compounded in these composite materials. These types of composite materials have already been put to practical use in various fields. However, it is difficult to synthesize the nanocomposite by this method.

In contrast, compounding is achieved by a process wherein the materials are synthesized by decomposing the pluralistic system material gas. In the in-situ CVD method, the composite material of type (II) can be synthesized. In this case, the disperse phase size of (a) to (d) becomes of the nanometer

order, and a nanocomposite is available.[4] The synthesis examples of CVD, ceramics, and nanocomposites that have been reported up to now are shown in Table 1.

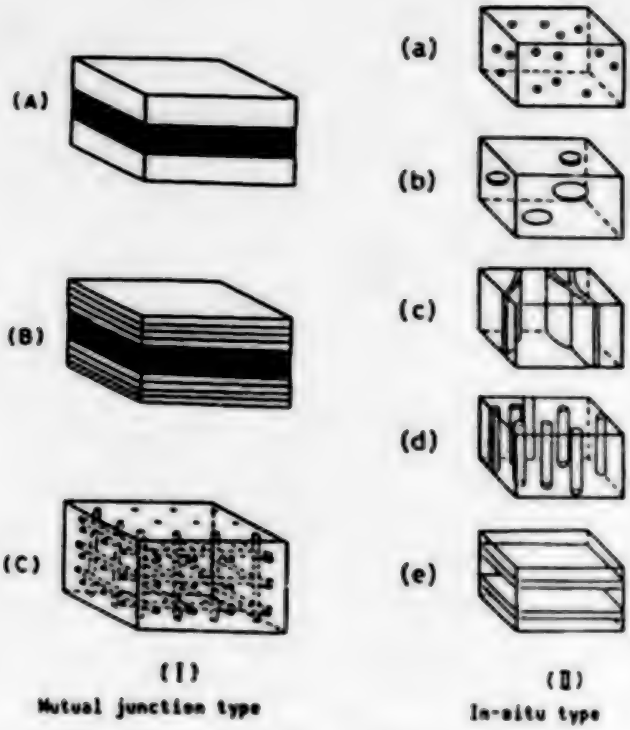


Figure 2. Type of Composite Materials

IV. CVD Fine Composites

Composite materials (fine composites) with new properties have been synthesized by considering elements other than materials as the disperse phase, i.e., by compounding (compositing) the elements. Those in which interest has been shown as disperse elements are shown in Table 2.

IV-1. Type of Disperse Phases

The disperse phase in a mother phase is a solid phase in conventional "composite materials," but a liquid phase and a gas phase (void) are also considered as the disperse phase in "fine composites." A gas phase such as a void or cavity is especially effective for improving the thermal insulating property and thermal stress relaxation characteristic. An example of this is a CVD-SiC/C system nanocomposite. Fine ceramics in which voids have been introduced can be synthesized by controlling the crystal growth direction (orientation) of SiC and controlling the C amount. This material excels in thermal insulating property. Moreover, the amount of voids can be controlled by utilizing the thermal expansion coefficient difference between the mother phase and the disperse phase.

Table 1. Synthesis Examples of CVD Nanocomposites

Mother phase	Disperse phase	Material gas	CVD temperature	Dispersion condition*
C	B_4C	$C_xHy-BCl_3$	1100 to 2000	a
	SiC	$C_3H_8-SiCl_4$	1440 to 2025	b
	TiC	CH_4-TiCl4	1200 to 2200	a
	ZrC	$C_xHy-ZrCl_4$	1300 to 1500	a
	HfC	$C_xHy--HfCl_4$	1300 to 1500	-
	BeO	$C_xHy-Be(CH_4O_4)$	1600 to 2000	a
BN	C	$BCl_3-NH_3-C_2H_2$	1700	-
		$BCl_3-NH_3-SiCl_4$	1400 to 1800	-
		$BCl_3-NH_3-TiCl_4$	1400	a
Si_3N_4	C	$SiCl_4-NH_3-C_3H_8$	1100 to 1300	a
	AlN	$SiH_4-NH_3-AlCl_3$	600 to 1100	-
	AlN	$SiCl_4-NH_3-AlCl_3-O_2$	-	-
	BN	$SiCl_4-NH_3-B_2H_6$	1100 to 1300	e
	BN	$SiCl_4-NH_3-BCl_3$	1400 to 1800	-
	TiN	$SiCl_4-NH_3-TiCl_4$	1050 to 1200	a,d
B_4C	C	$BCl_3-C_3H_8$	1400 to 1800	-
SiC	C	$SiCl_4-C_3H_8$	1300 to 1800	a
	B_4C	$SiCl_4-C_3H_8$-BCl3	1300 to 1800	-
	TiC	$SiCl_4-CCl_4-TiCl_4$	1300 to 1600	a
	Si_3N_4	$Si(CH_3)_4-NH_3$	1300 to 1500	-
ZrC	C	$ZrCl_4-CH_4$	1550 to 2100	-
Ti_3SiC2	TiC	$TiCl_4-SiCl_4$-CCl4	1000 to 1300	c
Ti-B-N	TiB_2	$TiCl_4-BCl_3-N_2$	1050 to 1500	a

*See Figure 2(II)

IV-2. Structure of Mother Phase and Disperse Phase

A "fine composite" consisting of a combination of a crystalline nimbostratus type structure and a noncrystalline structure can be considered from the standpoint of the mother phase and the disperse phase. Since a fine composite consisting of noncrystalline Si_3N_4 and nimbostratus type structure BN is transparent and does not crystallize even at a temperature of 1600°C, its application in window members for use at high temperature is being studied. Moreover, the thermal insulating property is improved by the increase in the conspicuous interface area in a fine composite in which noncrystalline Si_3N_4 and crystalline TiN is combined.

Table 2. Elements Considered in Complexing

Complexing element	Content
Forms of disperse phase:	Sphere, thin piece, thin layer, needle, fiber, column, (hollow)
Types of disperse phase:	Solid phase, liquid phase, gas phase (void, cavity)
Structures of mother phase and disperse phase:	Crystalline, nimbostratum structure, noncrystalline
Crystal perfection of crystalline mother phase and disperse phase	Defectless, lattice irregularity, laminate defect
Consistency and orientation of crystal when both phases are crystalline	Nonconformity, conformity
Interface of mother phase and disperse phase	Forming of reaction products and generation of stress by chemical reaction
Dispersion state of disperse phase	Uniform, nonuniform, continuous, discontinuous
Chemical bonding form of mother and disperse phase	Metallic bond, ionic bond, covalent bond, electronic bond

IV-3. Crystal Perfection of Crystalline Mother Phase and Disperse Phase

In a CVD-SiC fine composite, a lattice asymmetry and stacking fault are intentionally introduced by controlling the CVD conditions. A CVD-SiC fine composite in which the stacked fault synthesized at 1500°C has been composited has a high fracture toughness value.

IV-4. Consistency and Orientation of Crystals When Both Phases Are Crystalline

A nanocomposite in which fibrous TiN with a diameter of 10Å is dispersed in a B-Si_3N_4 mother phase that has been synthesized by the CVD method is a fine composite which has a close consistency of crystal structure between the mother phase and the disperse phase.

IV-5. Dispersion State of Disperse Phase

Fine carbon granules are dispersed in noncrystalline Si_3N_4 of the mother phase in a CVD-Si_3N_4/C system composite. When the C content of the disperse phase becomes more than 0.2 wt percent, it becomes a fine composite in which C exists in a netlike condition. The presence of electroconductivity appears to be due to this.

V. Synthesis of Functionally Gradient Materials by Complexing

So-called uniform functionality, in which the disperse phase exhibits homogeneous dispersion and the properties are uniform, as shown in Figure 2, was pursued in conventionally oriented composite materials. In recent years, however, the development of materials capable of withstanding more severe use environments has become necessary, and fine composites having a so-called "gradient function," in which the "elements" are continuously controlled and are not "uniform," such as those in Table 2, have come to attract attention. We are now working on creating an FGM by means of the synthesis of a gradient composition fine composite in which the composition is made continuously gradient from SiC to C by using the CVD method.

When $SiCl_4$ and C_3H_8 are used as the material gas, the flux of C_3H_8 gas is fixed and the amount of $SiCl_4$ vapor introduced is changed together with the synthesis time, and the CVD-SiC/C system graded composition fine composite is synthesized at a temperature of 1500°C. It is possible to introduce a microvoid at an optional position and to control the crystal orientation of the mother phase in this material. The sectional structure of this fine composite is shown in Figure 3.

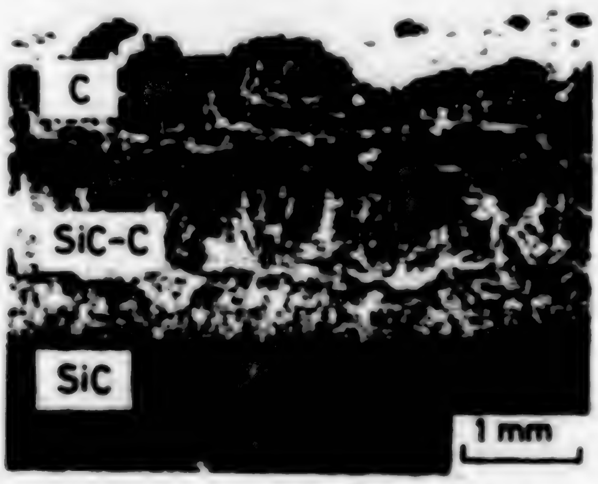


Figure 3. Sectional Structure of SiC/C System Gradient Composition Fine Composite Synthesized by CVD Method

VI. Conclusion

An effort has been made recently to obtain the compounding range in the synthesis of a CVD-nanocomposite by numerical computation, and material design is becoming possible in the synthesis field.

Moreover, material design has been attempted by the FGM research group on the functional gradient of materials in which the composition has been continuously controlled by the CVD method, and considerable results have been obtained.

In regard to fine composites that have been synthesized by complexing various elements by the CVD method, however, the effect exerted on the function of complexed elements has not been clarified theoretically. Further research is necessary in the future to apply the fine composite concept to the development of functionally gradient materials.

References

1. Masayuki Niino, Toshio Hirai, and Ryuzo Watanabe, JAPAN COMPOSITE MATERIAL SOCIETY JOURNAL, Vol 13, 1987, p 257.

2. Toshio Hirai, Masato Sasaki, and Masayuki Niino, MATERIALS, Vol 36, 1987, p 1205.

3. T. Hirai, International Forum on Fine Ceramics '88, Nagoya, March 1988.

4. Toshio Hirai, Inorganic New Fine Materials Introduction Seminar Text, Powder and Powder Metallurgy Association, 1981, p 63.

Preparation by Particle Method

43067596 Tokyo FGM NEWS in Japanese May 88 pp 14-16

[Report by Ryuzo Watanabe, Faculty of Technology, Tohoku University, on technology for the preparation of FGM by the particle method]

[Text] 1. **Raw Materials and Preparation Method**

The urgent topic now is the preparation of a superheat-resistant material of the thermal stress relaxation type. In the preparation of this material, heat-resistant ceramics is established on the high temperature side, metal or plastic is established on the low temperature side, and optimum composition control is established in the middle position. Dispersion of whiskers should also be considered for reinforcing the metal or plastic and for improving the toughness of ceramics. Thus, the raw materials to be used in the particle method are various metals and alloy powders, ceramics powders, plastic powder, and ceramic whiskers. The preparation technology basis is available from various existing element technologies in use in powder metallurgy and ceramics engineering. The simplest preparation process is shown in Figure 1. The material powder is prepared in the prescribed composition, mixed well, and the fluidity adjusted. Then, die molding or hydrostatic pressure molding is carried out by laminate filling or continuous composition control filling of the mixed powder into the die. This compact is either atmospheric sintered or pressure sintered. Microscopic composition control is achieved by adjusting the particle diameter and sintering condition of the material powder.

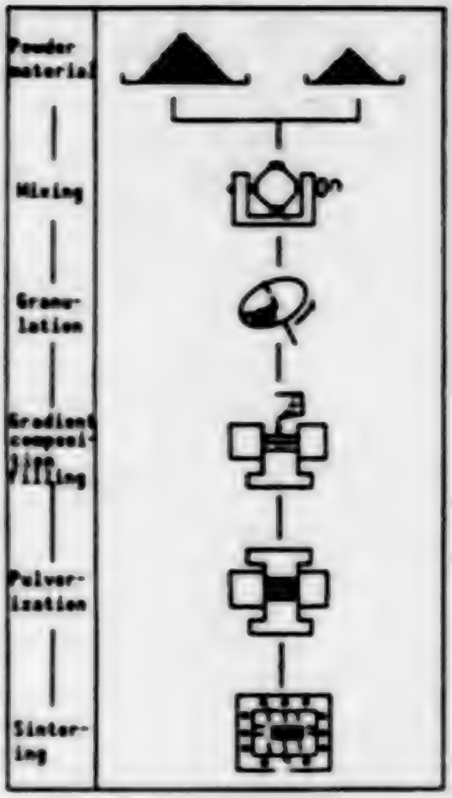


Figure 1. Preparation of Functionally Gradient Material by Particle Array and Sintering Method

2. Balance of Sintering Among Different Type of Powders

The first problem encountered when powders with different properties are mixed and gradient composition filling is carried out is the sintering balance of metal and ceramic powders. That is, in the sintering method used in the present situation, both types of powders must undergo the same sintered shrinkage at similar temperatures in order to obtain a sintered compact with a gradient provided in its composition. When there is a great difference in the sintered shrinkage amount of the mix ratio of metal powder and ceramic powder, the gradient composition control material becomes greatly strained. When there is an excessive difference, a crack generates locally and it is not possible to obtain a sound sintered material. Since the sintering temperature of ceramic powder is generally higher than that of metal powder, it is necessary to use a ceramic powder with an easy sintering property when carrying out sintering by the ordinary method.

3. Composition Distribution Design

Although a fine compact can be achieved by sintering the gradient composition control filler of metal powder and ceramic powder at high temperature, there is the fear of cracks generating in the compact interior due to the difference in the thermal expansion coefficient of the two materials during the cooling period from sintered temperature down to room

temperature. In fact, surface cracks are often observed in compacts in which a material design whereby the thermal stress generated is held sufficiently low during cooling from the sintered temperature in order to obtain a sound sintered gradient composition controlled material.

The following guidelines for thermal stress relaxation have been obtained as a result of conducting finite element analysis of thermal stress on a model in which an intermediate layer with a gradient provided in the composition has been introduced between the metal and the ceramic. (Article by Kawasaki and Watanabe in the JAPAN METAL SOCIETY JOURNAL, Vol 51, 1987, p 525.)

(1) Thermal stress concentration can be reduced considerably by introducing a gradient composition control layer between the metal and the ceramic.

(2) The finer the composition division of the gradient composition control layer, i.e., the smoother the composition control, the smoother the thermal stress distribution; however, the maximum value of thermal stress is not so dependent on the division fineness.

(3) The greater the thickness of the gradient composition control layer, the greater the thermal stress reduction, but the effect reaches saturation at a certain degree of thickness.

(4) The amount and distribution form of thermal stress are greatly dependent on the form of composition distribution. An optimum composition distribution exists for the thermal stress maximum value.

4. Survey on Relationship Between Composition and Various Characteristics of Nongradient Materials

To maintain sintering balance among different types of powders, it is necessary to know the relationship between the sintered shrinkage behavior and the composition. And to determine the optimum composition distribution for thermal stress relaxation, the relationship among the thermal expansion coefficient, the elastic modulus, the composition and the fracture strength level for each composite must be known. The composition distribution must be determined so that the thermal stress is sufficiently less than the fracture strength.

5. Trial Manufacture Examples (Zirconia/Stainless Steel System)

5.1 Molding and Sintering

[illegible] now, we have promoted the trial manufacture of gradient materials co[illegible]ining zirconia ceramics with various metals. Next, we will present an example of trial manufacture using a combination of ZrO_2-3 mol% Y_2O_3 (PSZ) and SUS 304 stainless steel.

PSZ powder with a nominal particle diameter of 0.05 µm and SUS 304 stainless steel with a mean particle diameter of 6 µm are mixed in a prescribed value between a mix ratio of 0 to 10 percent, die molded and at about 150 MPa,

then hydrostatic pressure molded at about 250 MPa. After laminate filling the mixed powders with compositions generally in accordance with suitable thickness and composition distribution, the gradient composition material was die molded and hydrostatic pressure molded as mentioned above. These green compacts were sintered at a temperature of 1350°C in a vacuum of 10^{-5} torr for 1 hour. In observing the sintering balance in this sintering system, we found that the sintering temperature for these materials in various compositions was approximately in the same range, but a slight difference was observed in the sintered shrinkage amount, as shown in Figure 2. However, it can be considered to be a good system for the sintering balance of metal powder and ceramic powder.

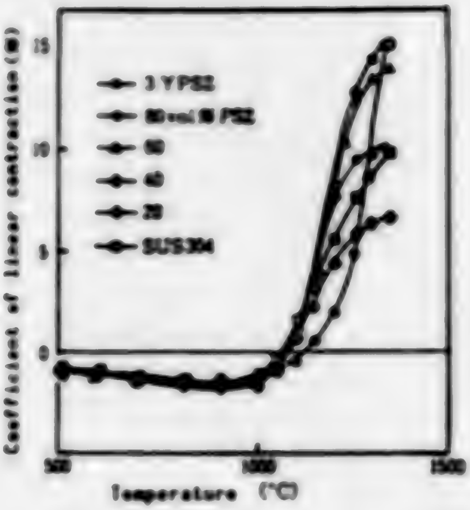


Figure 2. Balance of Sintering in PSZ/Stainless Steel System

5.2 Relationship Among Mix Ratio, Elastic Modulus, and Thermal Expansion Coefficient

The relationship among the elastic modulus, thermal expansion coefficient, and PSZ volume percentage in the PSZ/stainless steel system is shown in Figure 3. In contrast to the thermal expansion coefficient showing a change close to the linear rule, the relationship between the elastic modulus and the composition appears to be very irregular. This trend also appears in the relationship between the flexural strength and the composition, as shown in Figure 4. In the research conducted up to now, such a change has been closely related to the composition phase forming, and especially to the forming of the metal phase network (skeleton structure). The residual pores also secondarily control this relationship.

In any event, in the present state of composition design of gradient sintered materials, it is necessary to perform actual measurement of the elastic modulus and the flexural strength according to each mix ratio. After doing this, the thermal stress computation is carried out according to the basic principle of gradient composition control mentioned above, and

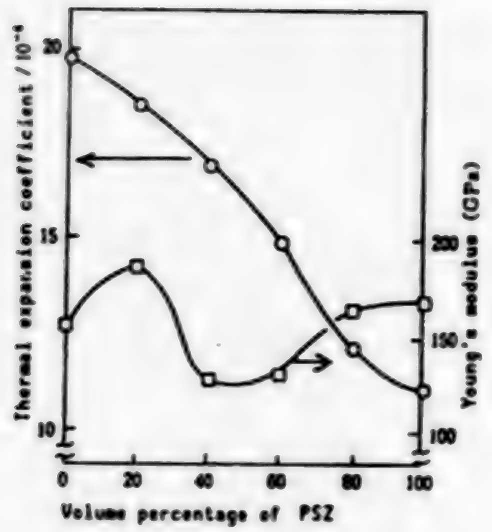


Figure 3. Relationship Among Elastic Modulus, Thermal Expansion Coefficient and Composition in PSZ/Stainless Steel System

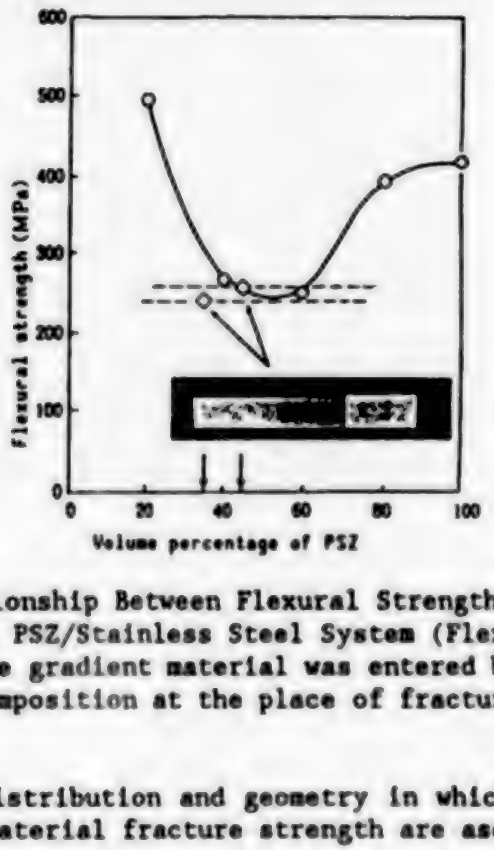


Figure 4. Relationship Between Flexural Strength and Composition in PSZ/Stainless Steel System (Flexural strength of the gradient material was entered by showing the composition at the place of fracture.)

thus a composition distribution and geometry in which the thermal stress does not exceed the material fracture strength are ascertained.

5.? Gradient Materials With Optimum Composition Distribution

The optimum composition distribution in this system obtained from a finite element computation based on various measured values of a nongradient

material is shown in Figure 5. In it, the thermal stress is sufficiently lower than the flexural strength value.

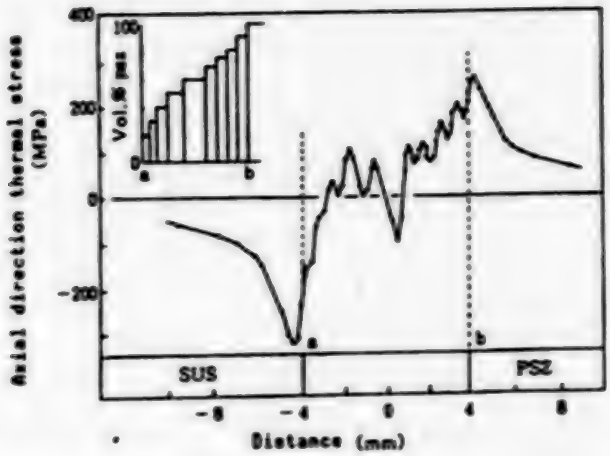
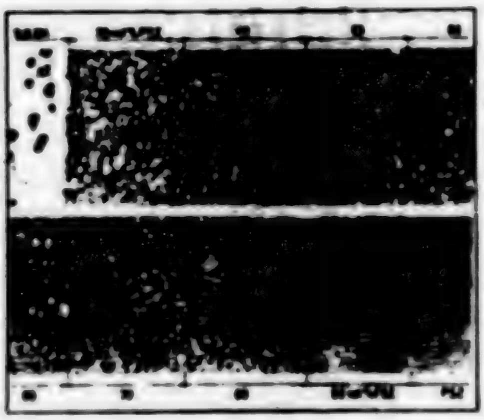


Figure 5. Optimum Composition Distribution in PSZ/Stainless Steel System and Thermal Stress Distribution Vis-a-Vis the Optimum Composition Distribution



Photograph 2. Microstructure of PSZ/Stainless Steel System Gradient Composition Control Material

Photographs 1 [not reproduced] and 2, respectively, show a bending testpiece and the microstructure of a gradient composition control material cut out from a cylindrical sample. As shown in these photographs, there are no large macroscopic or microscopic defects in the gradient material. The bending test data of the testpiece shown in Photograph 1 are used in Figure 4, wherein a fracture has been generated at approximately the place of minimum strength composition discovered in the nongradient material. This fact shows that the local strength of the gradient composition control material corresponds well with the nongradient material strength of the same composition, and it can be said that the condition of preparation of the test has been appropriate.

6. Conclusion

The functionally gradient materials presently obtained using the particle array method are a combination of zirconia ceramic and several types of metals. The shape is simple, and the composition of the material has been changed in one direction only by staged composition control. It is my view based on experience up to now that the development of a powder filling array technology and new sintering technology are necessary to make it possible to obtain diverse metal and ceramic combinations, complex shapes, fine composition control, and three-dimensional composition control.

Research on Design Support System

43067596 Tokyo FGM NEWS in Japanese May 88 pp 17-18

[Report by Tetsu Hirano, Daikin Industries, Ltd., on research on computer-aided FGM design support system]

[Text] Our company has been in charge of this topic since 1987 as a member of the Materials Design Subcommittee in the "research on the basic technology of functionally gradient materials development for thermal stress relaxation" conducted under the science and technology promotion adjustment expenditure category. The research is aimed at: 1) development of an FGM design support system, and 2) verification of this design theory using plastic functionally gradient materials. An outline of the contents of this research is presented here.

(1) Development of Prototype for FGM Design Support System

The important thing in the design of FGM is to determine the optimum material combination and internal composition distribution for thermal stress relaxation. When approaching this problem from a different standpoint, we find that it is an inverse problem wherein the physical property value (internal composition) distribution is determined on the basis of the final use condition (boundary condition) of the material. Or again, in a case wherein the function called thermal stress relaxation has been manifested in the material itself, material that is conventionally treated as a structural member, it can be called functional design and structural design by integration. We have taken such an approach under the concept of "inverse design" and have studied the technology for its systemization.[1,2] As a result, an optimum design was achieved by the use of the thermoelastic composition concept (Tokyo Institute of Technology in charge) based on the experimental mixing rules on the physical properties of a material (Table 1), the use of a macrostructure model of the polyphase mixing system, and the use of a one-dimensional FGM thermal stress analysis technique (National Aerospace Laboratory in charge). At the same time, a prototype of an FGM design support system that graphically displays the analysis and inference processes was realized in the inference language Prolog.[3,4] This prototype, the core of which is the inference system "D^{-1} (inverse design)--Prolog" based on the inverse design theory, consists of a workstation in which are integrated a physical property estimation module

and a one-dimensional thermal stress analysis module that uses Fortran language, an easy database that uses Prolog and a graphic interface that uses C language.[4] An example of the picture display is shown in Figure 1. The inference mechanism of this system is able to identify the microstructure and rule of mixing from the physical property measured values of nongradient and gradient materials and is also utilized in the verification of the design theory, which will be discussed later.[2,4]

Table 1. Selection of Mixing Rule by Microstructure

Phase array in virtual layer [Blue: continuous phase White: Disperse phase]	Laminated	Fiber(1)	Fiber(2)	Thin Layer	Flake-like	Spherical Particle
Thermal conductivity λ	Harmonic mean rule	Harmonic mean rule	Linear composite rule	Linear composite rule	Kerner mixing rule	Kerner mixing rule
Thermal expansion coefficient α	Rigidity adjustment type composite rule	Rigidity adjustment type composite rule	Linear composite rule	Linear composite rule	Eshelby mixing rule	Kerner or Turner rule
Elastic modulus E, K, G	Linear composite rule	Linear composite rule	Harmonic mean rule	Harmonic mean rule	Eshelby mixing rule	Kerner mixing rule

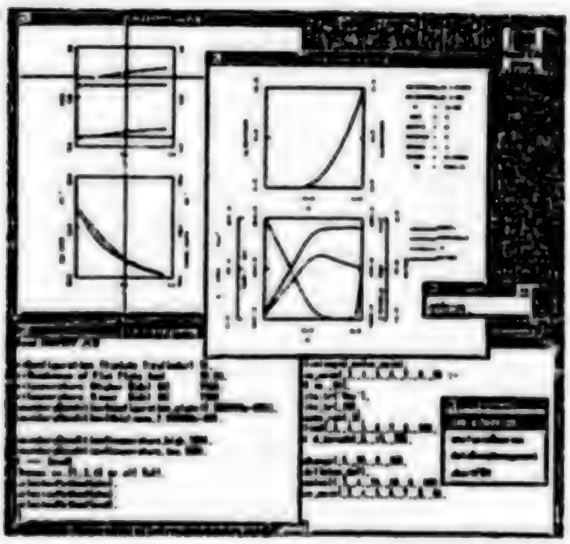


Figure 1. Functionally Gradient Materials Design Support System

This system has been used for the primary design of rocket combustors and space shuttle thermal protection materials combining several types of materials.[3,5]

(2) Synthesis of Plastic FGM and Verification of This Design Theory

Adjustment and physical property measurement of a nongradient material in which PTFE (polytetrafluoroethylene) was combined with bronze or silver were carried out in order to develop a plastic model of FGM. A two-component system plastic staged FGM consisting of PTFE and silver was synthesized using four different types of compositions by means of the particle array method, and their temperature distribution and heat flux were measured using the simplified temperature head testing device of the National Aerospace Laboratory. By means of an inverse computation of the gradient composition distribution parameter by entering the obtained measured values in the inference system mentioned previously, the initial design values could be identified with good precision when the physical properties of the gradient materials were determined by interpolating the actual measured values of nongradient materials. However, the design values of the gradient composition distribution parameter could not be identified when the physical properties of the gradient materials were determined by the rule of mixing. It is considered that this was because micropores had generated in the microstructure of the plastic gradient material, and especially in parts with a high volume percentage of silver.

As future research topics, an interface with the FGM database that is to be gradually built up by the National Aerospace Laboratory will be developed, together with the addition of functions to the prototype of the design support system. Moreover, the system will be constituted so that a third component--the effects of material nonlinearity and micropores--can be taken into consideration in the thermal stress analysis function. Also, plastic FGM containing micropores will be synthesized, and the third component system design theory will be verified by conducting measurements on temperature distribution and thermal stress.

References

1. Hirano, Sumiyoshi, Niino, and Sata, "Inverse Design Approach to Material Design of Functionally Gradient Materials," Machinery Society, Lecture on Strength of Materials, No 860-10, 1986, p 21.

2. Hirano, Sumiyoshi, Yamada, and Niino, "Attempt at AI Approach Application to Inverse Problem Analysis," Machinery Society, Lecture on Strength of Materials, No 870-12, 1987, p 20.

3. Hirano, Sumiyoshi, Ishikawa, Wakamatsu, Watanabe, and Sata, "Basic Research on the Application of Functionally Gradient Materials to Rocket Combustors," Manuscripts of Lectures for 31st Space Technology and Science Joint Lecture, 1987, p 628.

4. T. Hirano and T. Yamada, "Multipradigm Expert System Architecture Based Upon the Inverse Design Concept," Proc. of Int. Workshop on Artificial Intelligence for Industrial Applications," Hitachi, Japan, May 1988.

5. T. Hirano, T. Yamada, J. Teraki, M. Niino, and A. Kumakawa, "A Study on a Functionally Gradient Materials Design System for a Thrust Chamber," 16th Int. Symp. on Space Technology and Science, Sapporo, Japan, May 1988.

Wide Area Synthetic Molding Technology

43067596 Tokyo FGM NEWS in Japanese May 88 pp 19-20

[Report by Nobuhiro Sata, Government Industrial Research Institute, Tohoku, on research on wide area synthetic molding technology under hydrostatic pressure by the self-exothermic reaction method]

[Text] Materials synthesis in a wide area is a prerequisite when considering the use of functionally gradient materials as a functional coating on a metal surface. There must not be a "joint" that exerts a bad effect as a defect in the material in the area of this materials synthesis. Such a "joint" may become a fatal defect for FGM. Thus, FGM must be made as one body when it is to be synthesized in a wide area. A patchwork is not appropriate for FGM.

One of the research topics on FGM of the Government Industrial Research Institute, Tohoku, is to ascertain whether this requirement can be met by using the self-exothermic reaction method. The means to do this is clear: what must be done is to conduct various confirmatory experiments until the target is reached, eliminating any small problems lurking in the process. I will now discuss briefly the basic conditions which should be taken into consideration in using the self-exothermic reaction method, and the outlook for practical achievement of so-called wide area control technology.

The characteristic of the self-exothermic reaction method is that synthesis is propagated with a directional property in an extremely short time. This composition has both advantages and disadvantages. Up to now, we have conducted research on simultaneous synthesis molding technology using a very convenient uniaxial pressure method (spring compression method) that utilizes spring pressure. This is a good, efficient method for matching the propagation direction and pressurizing direction of the reaction. However, while this method is all right for a simple shape such as that of a cylinder, when considering the manufacture of coatings over a wide area and compacts of complex shapes, the uniaxial pressure method loses its effect; it is not suitable for wide area synthesis. A method of actuating isotropic compressive pressure is necessary. Therefore, we arrived at the method of synthetic molding under hydrostatic pressure using water as the pressure medium as a means that also takes economy into consideration.

Figure 1 shows a typical method of wide area synthetic molding under hydrostatic pressure. The device used is an autoclave that has an electrode terminal for ignition and is capable of raising the temperature and pressure. The pressure vessel that is presently being used for experiment has an inner diameter of 9 cm, a height of 23 cm, an inner capacity of about 1.5 liters, and is capable of raising the temperature and pressure up to the maximum of 350°C and 30 MPa, respectively. After vacuum sealing of the

material powder together with the ignition jig in a sealed container made of copper, it is suspended in water. An accumulator is provided to compensate for the small compressibility of water and to maintain the pressure approximately at a certain level according to the shrinkage of water at the time of synthesis so that the gas in the accumulator is always at pressure balanced with the water. Furthermore, the heat of reaction escaped into the water through the copper mold and it may be that the synthesized part will be gradually refined at the time of synthesis by means of the flexible metal wall. The electrode for ignition is led out to the autoclave exterior by a lead wire and the material in the sealed vessel is ignited by applying electric current instantaneously to the ignition jig. A plate-shaped synthetic sample, as shown in Figure 2 [not reproduced], is available by this method.

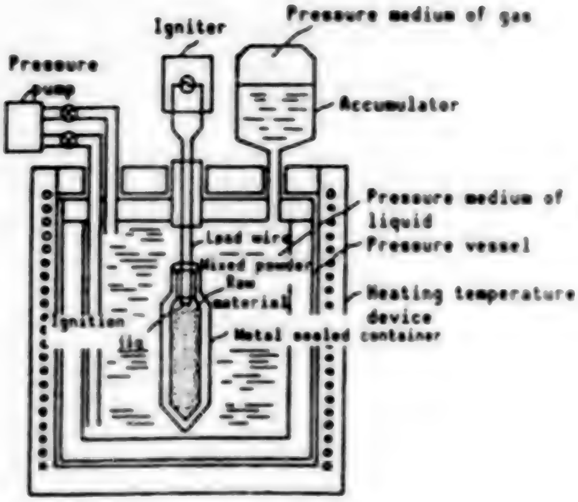


Figure 1. Method of Wide Area Synthetic Molding Under Hydrostatic Pressure

The merit in using water pressure is that a comparatively cheap device can be made, whereas this would be quite difficult using gas pressure. It is also less risky, as the compressibility of water is small. Since water has a high heat capacity, it operates as a coolant in the synthesized sample. It serves to provide a thermal insulation if the cooling effect is too great. Since the wall of the metal vessel (copper is considered suitable) does not melt, there is no fear of it mixing with the synthesized material.

Some points to be taken into consideration when using hydrostatic pressure are that the vessel itself must have a heat capacity high enough that it does not melt, and at the same time, it must be easily deformed following shrinkage at synthesis. Also, as this method differs from the spring compression method in that the direction of reaction propagation does not coincide with the pressure direction and they are perpendicular to each other, it is sometimes considered inefficient for fining. However, it is possible to control the reaction propagation speed by the composition and density of the material, and the reaction propagation direction and pressure direction can be matched by deformation of the reaction interface of the

material interior. Thus, it is possible to form coating surfaces having various shapes. Based on these prospects, we plan to prepare further large-sized test equipment in 1988. With this equipment it may be possible to extract the problems in realizing wide area, large-scale synthesis and find the means to solve them.

Ultrahigh-Temperature Fracture Evaluation

43067596 Tokyo FGM NEWS in Japanese May 88 pp 21-20

[Report by Kazumi Hirano, Mechanical Engineering Laboratory, on research on ultrahigh-temperature fracture strength characteristic evaluation of FGM]

[Text] **Introduction**

"Research on basic technology of functionally gradient materials development for thermal stress relaxation"--that is the FGM national project started on a full scale in 1987 under the category, science and technology promotior adjustment expenditure. This national project aims at the creation ot superheat-resistant and thermal protection materials in support of the concept of Japan's development of space shuttles and spacecraft. I will present two or three of the recent mechanical strength evaluation technology research results achieved as part of the R&D on characteristic evaluation technology of FGM, of which the Mechanical Engineering Laboratory has been in charge.

The aim of this research lies in ascertaining the microscopic fracture mechanism of FGM under ultrahigh temperatures and in establishing a fracture strength characteristic overall evaluation method through the development of damage analysis and monitoring technology. A further aim is to develop FGM excelling in high-temperature strength and fracture toughness by actively providing feedback of the results to those conducting research on materials design and structure control technologies, together with ascertaining the effects of the microstructure factors exerted on strength and fracture toughness (including fracture resistance).

The research program and contents for this year aimed at the extraction of the microstructure factors controlling the fracture strength characteristics of microstructure control uniform functional materials are: 1) development and maintenance of the material strength gradient characteristic evaluation equipment made in conjunction with the trial manufacture of an ultrahigh-temperature furnace whereby atmosphere control (argon, nitrogen in a vacuum (10^{-5} torr)) can be achieved; 2) implementation of flexural and fracture toughness tests of microstructure control uniform functional materials; and 3) fracture mechanics and fractographic studies on correlation of strength, fracture toughness and microstructure factors. An outline will be presented of the fracture toughness K_{IC} test using the developed atmosphere-controlled ultrahigh-temperature furnace and the uniform functional material TiB2-Cu by the IF (indentation fracture) method as part of the FGM fracture strength characteristic evaluation research under the ultrahigh technology of $\leq$2,000°C.

Development of Atmosphere-Controlled Ultrahigh-Temperature Furnace

The appearance of the atmosphere-controlled ultrahigh-temperature furnace that has been trial manufactured is shown in Figure 1 [not reproduced]. Strength and fracture toughness tests by three-point and four-point bending are possible under the ultrahigh temperature of up to 2000°C by attaching this furnace to existing material testing equipment. Fracture toughness tests, etc., are presently being conducted by the CN (chevron notch) method intended for fiber reinforced composite materials, in which uniform functional materials such as glass, ceramics and carbon are used as the matrix, and the microstructure factors that control strength and fracture toughness under ultrahigh temperature are extracted.

Fracture Toughness Test by IF Method

An example of the fracture toughness test result on TiB_2-Cu using the IF method is shown in Figure 2 [not reproduced]. TiB_2-Cu is a material that has been synthesized by the Government Industrial Research Institute, Tohoku, which sought in this project to synthesize an FGM in the ceramics-metals system using the self-exothermic reaction method. This is a uniform function material in which the weight ratio of TiB_2 and Cu is 1:1.

Microcracks generated by Vickers indentation are caused by anisotropy and are bent. The interaction of the residual stress caused by the indentation pressure and that caused reaction sintering also play a part in this. The hardness resulting from the nonuniformity of the microscopic structure (TiB_2 and Cu phases) and the fluctuation of K_{IC} (2 to 12 MP $\sqrt{m}$) are great. Moreover, many microcracks emanating from pores that are thought to be generated in the reaction sintering process are also observed. A tendency is observed for these microcracks to progress along the interface of the TiB_2 and Cu phases resulting from residual stress, etc., caused during manufacture by the difference in thermal expansion coefficient. This differs from the aspect of cracks progressing as a result of Vickers indentation.

A study is now underway to ascertain the characteristics of TiB_2-Cu, including the microcracks generated in the reaction sintering process, which are regarded as essential to the success or failure of FGM synthesis of the ceramics-metals system by the self-exothermic reaction method.

Outline of Joint Subcommittee

43067596 Tokyo FGM NEWS in Japanese May 88 p 22

[Report on meeting of joint subcommittee on promoting research on basic technology of FGM]

[Text] The joint subcommittee on promoting "research on basic technology of FGM for thermal stress relaxation" was convened for 2 days, 14 and 15 March, at the Aoba Memorial Hall, Faculty of Technology, Tohoku University. After a welcome by Chairman Koizumi, announcements of the results obtained were made by the respective organizations on structure

control, material design and characteristic evaluation, followed by a spirited question-and-answer session. A social gathering was held on the evening of the first day, with participants enjoying refreshments and a pleasant chat.

Structure Control Subcommittee

43067596 Tokyo FGM NEWS in Japanese May 88 pp 22-23

[Report by Ichiro Shioda, National Research Institute for Metals, on the work of the Structure Control Subcommittee]

[Text] The following reports were given by the various research agencies in this division, accompanied by a question-and-answer session after each report.

I. Physical and Chemical Vapor Deposition Methods

1. Physical Vapor Deposition Method (National Research Institute for Metals)

NFGM's (new functionally gradient materials) of various compositions can be achieved in the Ti-TiN, Cr-CrN, and Ti-TiC systems by control of the reaction gas flux. Moreover, the prospect of forming functionally gradient materials by continuously changing the gas flux is also envisaged.

Question: Isn't there peeling in the case of thick film deposition?

Answer: There is no peeling when a metallic coating is applied to the substrate and NFGM and FGM are formed on top.

Question: What are the x and y values of Ti_xN_y?

Answer: They have the stoichiometric ratio of x = 1 or 2 and y = 1.

2. Chemical Vapor Deposition Method (Metal Materials Laboratory, Tohoku University)

NFGM plate samples were available over the entire composition range of SiC/C by controlling the temperature and pressure. Although the thermal expansion coefficient of CVD-C is small in the C-axis direction, the thermal expansion coefficient is improved when SiC has been subjected to eutectoid reaction.

Question: What is the method of measuring thermal expansion, and what is the size of the sample?

Answer: Measuring is accomplished by the laser flash method, and the size of the sample is 10ϕ x 0.7 - 1 mmt.

Question: How is adjustment made in the composition ratio of C(CVD) → SiC(SiC)?

Answer: Adjustment is made as occasion demands

3. Physical and Chemical Vapor Deposition Fusion Methods (Sumitomo Electric Industries, Ltd.)

Thermal cracks were generated in SiC film formed on the C/C composite by the CVD method, and the oxidation resistance deteriorated in correspondence with the cracks. No cracks were observed when the C/C composite was coated with TiN.

Question: What are the roles of CVD and PVD in the fusion method?

Answer: It is preferable to use the material obtained by PVD on the low temperature side and the material obtained by CVD on the high temperature side.

Question: What surface treatment is used for the C/C composite?

Answer: The surface treatment consists only of machine polishing.

II. Research on Structure Control Technology by Particle Method

1. Particle Injection Method (Material Processing Course, Faculty of Technology, Tohoku University)

The sintering of SUS 304, Mo, Ni powders and ZrO_2-3 mol% Y_2O_3 powder was possible in all mix ratios. The thermal expansion coefficient of a mixed sintered material is close to that of the linear mix rule. The Young's modulus and flexural strength in the case of the ZrO_2-SUS system is 40 - 60 vol%, and the minimum value is adopted. Moreover, the composition distribution for thermal stress relaxation was analyzed by means of FEM, and the conditions for obtaining gradient sintered materials with a sound layer structure were ascertained.

Question: What is the black part seen in the FGM microstructure? And what contribution does it make to the characteristics of the structure?

Answer: The black part is a pore. It does not contribute to the thermal expansion coefficient, but the Young's modulus is lowered.

2. Research on Laminate Array Technology by Thin Filming of Particles (Nippon Kokan K.K.)

The appropriate mix ratio and drying condition of the sheet in the production of green sheets of ceramics and Ni powder using a binder and plasticizer have been ascertained. It has also been ascertained that the optimum sintering temperature is 1550°C for ceramics and 1350°C for metals after degreasing. Moreover, the prospect is envisaged of producing FGM by the Ni-ZrO_2 composite plating method.

Question: What is the minimum thickness of a sheet and the maximum number of laminates?

Answer: The minimum sheet thickness is about 20 μm, and a maximum of 100 laminates is possible.

Question: What is the maximum Vf of a composite plating?

Answer: It is freely controllable in the range of up to a maximum 30 percent.

III. Research on Laminate Molding Technology by Thermal Spraying Method

1. Independent Technology for Thermal Spraying of Different Types of Particles (National Research Institute for Metals)

It has been ascertained that the conditions for thermal spraying of Ni-Cr are stable over a wide range, while those for thermal spraying of ceramics are superior under large current and high gas flux.

Question: What is the method of measuring film tensile strength, and what is the measured value?

Answer: Thermal sprayed film of 30ϕ x 0.3 is pulled in a perpendicular direction to the film surface and measured. The tensile strength is about 30 MPa in metals and about 11 MPa in ceramics. (Comment: The tensile strength of 40 MPa is available in pressure reduced thermal spraying of PSZ and the tensile strength of 60 MPa is available in HIP.)

Question: What is the frame interference when using two spray guns?

Answer: Studies will be made on interference during 1988.

2. Technology for Simultaneous Thermal Spraying of Different Types of Particles (Nippon Steel Corp.)

Metal and ZrO_2 were independently thermal sprayed with thermal spraying guns of different stable operating pressure reduction ranges, and stable thermal spraying was possible in the range of 50 - 350 torr. Studies are now being conducted on the conditions for simultaneous thermal spraying.

Question: What is the thermal spraying thickness, and what is its homogeneity?

Answer: It differs according to the substrate, but the thickness is a little less than 1 mm. Thermal spraying of three paths has been conducted, and it was homogeneous at 30ϕ under the optical microscopic level.

IV. Research on Structure Control Technology by Self-Exothermic Reaction Method

1. Reaction Control Technology (Industrial Science Laboratory, Osaka University)

The combustion propagating speed of TiB_2 becomes rapid with the rise of the sample filling density, but the combustion temperature does not change. It can be considered that the combustion temperature of TiB_2-Cu has reached the melting point of TiB_2 when Cu is contained up to 20 percent. The combustion propagating speed decreases in accordance with the amount of Cu added.

Question: What is the melting point of TiB_2, and does any Ti remain after the reaction?

Answer: The melting point of TiB_2 was 2900°C in the measurement conducted at this time; no Ti remained after the reaction.

2. Wide Area Control Technology (Government Industrial Research Institute, Tohoku)

The thermal expansion coefficient of TiB_2-Cu system nongradient materials of various compositions increases approximately in proportion to the Cu concentration. Moreover, the thermal expansion coefficient change is slight up to the Cu weight concentration of 50 percent, and it suddenly becomes large after exceeding this limit.

Question: What is the porosity and oxidation resistance of TiB_2?

Answer: The porosity becomes minute with more than 50 percent of Cu. Such means can be used as 1) surface coating, 2) substituting ZrB_2 for TiB_2, and 3) replacing Ti with Ni and B with C.

Characteristic Evaluation Subcommittee

43067596 Tokyo FGM NEWS in Japanese May 88 p 24

[Report by Hideaki Takashi, Faculty of Technology, Tohoku University, on the work of the FGM Characteristic Evaluation Subcommittee]

[Text] The evaluation group conducted studies focusing on the development and enhancement of an evaluation test method aimed at developing the technology for evaluating the materials characteristics of FGM using the machinable ceramics (trade name, Macoru) adopted as a standard sample. In 1987, several types of nongradient materials were selected for joint research with the structure control group. The main contents reported for each research item are as follows:

(1) Technology for Quantitative Evaluation of Local Thermal Stress (Ship Research Institute, MITI)

Studies were conducted on a technique of analysis of local thermal stress in which laser ultrasonics and the CT method were combined. An algorithm was devised for evaluating the temperature distribution and thermal stress distribution from the sample transmission time data of ultrasonics.

(2) Thermal Insulation Performance Evaluation Technology (Sumida Branch of National Aerospace Laboratory)

Reports were made on the state of preparation of high-temperature head basic evaluation test equipment aimed at providing a high-temperature head for FGM. This equipment consists of a 30 kW Xe arc lamp for heating, a liquid hydrogen supply pipe for cooling, and a vacuum bell jar for maintaining the sample in a vacuum atmosphere. A thermal insulation performance evaluation experiment is possible under the conditions of a maximum temperature head of 1000°C and a maximum thermal flux of 5 MW/m^2. Also, measurement of the Macoru thermal conductivity was conducted by the propane combustion gas heating method (stationary method) as a simulated actual environment basic test on a high-temperature gas flow site, and it was shown that it coincided with the following results obtained using a nonstationary method.

(3) Technology for Evaluation of Thermal Physical Property Values (Sumida Branch of National Aerospace Laboratory and Energy and Mechanical Engineering Course, Faculty of Technology, Shizuoka University)

Evaluation of thermal conductivity and thermal diffusivity using the stepped heating and pulsed heating methods and the measurement of specific heat by the ASC method were conducted and the results obtained on the Macoru and SUS-PSZ samples were presented. A computation program to obtain the thermal diffusivity of a three-layer sample was developed.

(4) Technology for Overall Evaluation of Thermal and Mechanical Characteristics

(4.1) Thermal Fatigue Evaluation Technology (Nagoya Aircraft Manufacturing Plant, Mitsubishi Heavy Industries, Ltd.)

A temperature cycle was installed in an infrared image furnace containing a uniform temperature field, with the Macoru sample and SiC whisker·A alloy as the objects. Then, thermal fatigue evaluation was made by measuring the three-point flexural strength. The fatigue strength in the Macoru sample increased together with the temperature cycle, while it dropped in the SiC whisker·A alloy when the temperature cycle exceeded 100 cycles.

(4.2) Thermal Shock Evaluation Technology (Material Strength Research Institute, Faculty of Technology, Tohoku University)

An SP (small punch) method using an ultrasmall testpiece for fracture strength evaluation and an MSP method enabling high-precision displacement measurement were developed. Through these developments, evaluation of the

fracture strength, including such mechanical characteristic values as the Young's modulus, in brittle and ductile materials has been made possible by a single testing procedure. Moreover, a thermal impact test was conducted on the Macoru and PSZ plasma coating material using the CO_2 laser, and the corresponding correlation between thermal impact crack generation and AE emission behavior was studied. It was found that the time of crack generation could be detected by measuring the AE (acoustic emission) signal, and a quantitative evaluation could be made by measuring the laser critical output density at this time, so it was proposed to applying this method in the evaluation of the thermal impact characteristic of various functionally gradient materials.

(4.3) Mechanical Strength Evaluation Technology (Mechanical Engineering Laboratory, Agency of Industrial Science and Technology, MITI)

Development was carried out of material strength characteristic evaluation equipment with a maximum temperature of 2000°C and capable of atmosphere control. Also, it was proposed to adopt the chevron notch method and the indentation method as means to evaluate fracture toughness values.

Design Subcommittee

43067596 Tokyo FGM NEWS in Japanese May 88 p 25

[Report by Tetsu Hirano, Daikin Industries, Ltd., on the work of the FGM Design Subcommittee]

[Text] Reports were made by the following organizations on the state of progress of research in the design area.

(1) "Research on Physical Property Data Estimation Technology for Designing Functionally Gradient Materials" (Precision Engineering Laboratory, Tokyo Institute of Technology]

The thermoelastic architectural theory of the polyphase mixing system was adapted from the micromechanics standpoint, and a computation method was used wherein the elastic constant and thermal expansion coefficient was formularized for theoretically estimating the physical properties necessary for the design of FGM. In particular, the unified expression of various microstructures was possible using a rotary ellipsoidal model.

(2) "Research on Development of Functionally Gradient Material Theoretical Model and Thermal Stress Analysis Technique" (Sumida branch of National Aerospace Laboratory)

The technique of thermal stress analysis of functionally gradient materials was formularized by using an infinite plate and steady heat conduction model, and a thermal stress minimizing design was created on the basis of the combination of three types of systems (SiC-C system, ZrO_2-W system, ZrO_2-Mo system). Also, the thermal stress when producing the SUS-PSZ system by the sintering method was analyzed.

(3) "Research on Computer-Aided Functionally Gradient Materials Design Support System (Daikin Industries, Ltd.)

A prototype of an FGM expert design support system having the three functions of design, analysis and database control was developed. In particular, inverse analysis based on experimental data has been made possible as an analysis function together with regular analysis. Moreover, the design technique has been verified by an actual temperature measurement made on plastic FGM.

(4) "Research on Measurement and Evaluation of Thermal Physical Property Values of Functionally Gradient Materials" (Faculty of Technology, Shizuoka University)

A measurement experiment was conducted using the standard sample Macoru for establishing the thermal physical property value measurement method appropriate to functionally gradient materials. The stepped heating method and pulsed heating method of thermal diffusivity measurement were compared. Moreover, the thermal diffusivity measurement method for three-layer material was studied.

(Questions and Answers)

Active discussions were conducted on these reports, and the gist of the questions and answers is as follows:

(1) The present state of design and analysis is one-dimensional and a boundary temperature is designated. However, two-dimensional and three-dimensional analyses will be necessary in the future. Interface heat transfer should also be taken into consideration.

(2) There should not be haste in narrowing down the selection of FGM material combinations; rather, the possibilities should be widely explored.

(3) In regard to the peculiar behavior of physical property values (Young's modulus, etc., of the SUS-PSZ system) in the intermediate mixing phase of FGM, studies will be conducted on the actual measured value base design and on a method taking into consideration pores in the theoretical physical property model. Moreover, studies will be conducted on whether a plastic FGM can be produced wherein the shape and distribution of pores can be artificially controlled.

(4) What is the evaluation of the difference in the stationary method and the nonstationary method in measuring the thermal physical property? Initially, there should be no difference as a result of measurement methods.

(5) It is anticipated that difficulty will be encountered in measuring the thermal physical property of a specimen of some several tens of microns.

(6) Isn't measuring the thermal physical properties of FGM in bulk meaningless? For example, even when considering the overall heat transfer coefficient as the amount corresponding to thermal conductivity, this changes depending on the internal composition distribution of FGM.

20158/9365

Construction Company Enters Biotechnology Field

43066558 Tokyo ZAIKAI TEMBO in Japanese Aug 88 pp 112-120

[Article by reporter Shudan Toprai: "Construction Company Also Rushes Into Bioindustry"]

[Excerpts] Taisei Corporation, a large general construction firm in Japan, established a "Biotechnological Institute" this March. Strictly speaking, it serves as a satellite institute to the firm's Technical Research Institute, but it is reportedly conducting microbiological, marine biotechnological and plant-related biotechnological research. The question, "What does biotechnology have to do with a construction company?" has been raised.

Biotechnology Related to Reproduction Technology Hits Its Stride

On 16 March 1988, a research institute with a large farm came into being on a piece of reclaimed land facing Tokyo Bay in the city of Narashino, Chiba Prefecture. It is the Biotechnological Institute of Taisei Corporation.

This entire area is expected to be developed hereafter as a research town. There are still many vacant lots, and notice boards reading "Land Set Aside for Development" are drawing attention. However, now that the JR Keiyo Line also has become operational, a new town may possibly take shape here in the near future.

"Why do all the enterprises become involved with biotechnology?"

As if to answer that question, Seiji Kaneko, who took office as the first chief of the new institute, says: "Resources are not inexhaustible. However, biotechnology always involves reproduction technology, and this can even be referred to as one of its characteristics. Precisely for that reason, many enterprises are investing heavily in it."

It was in 1985 that this company first became involved with biotechnology. It began with a biotechnological laboratory in its Technical Research Institute located in Totsuka Ward, Yokohama City. As early as 1986, the following year, the idea of establishing a new institute emerged. On 1 April of this year, the firm carried out a drastic reform of its research organization, and the biotechnological laboratory became the Biotechnological Institute by merging it with other related sectors. Prior to the reorganization, transfer to the new institute had begun in March.

While it is apparently an independent institute, strictly speaking it is an appendage of the Technical Research Institute and is positioned as a satellite institute. It can probably be said that this company expects much of the biotechnological sector and that this sector has started full-scale operations.

There may be many who wonder, "What does the construction industry have to do with biotechnology?" But it has been involved in biotechnology-related matters before. It was, however, in 1985, as mentioned above, when a laboratory was created especially for this purpose.

When the firm decided to participate in biotechnology on a full scale, the question was how to deal with it. Its experience was insufficient to the point of being nonexistent.

Commenting on the state of affairs at that time, Deputy Chief Heiichiro Okawa of the Technical Research Institute says: "What is first considered when starting a branch where there is no accumulation of technology is to invite researchers from somewhere else. And then, there is a way of purchasing them together with their institute. This company, however, did not do so. It took measures to establish a laboratory by collecting engineers from branches having some connection with biotechnology."

The development plan for this institute can be viewed in terms of a three-step process.

"First, initial measures are taken to acquire various technologies already open to the public. This is the first step. To push research into a new field on the basis of the technologies acquired is the second step. The third step, of course, is biotechnology on a full scale" (Deputy Chief Okawa).

He says that the company is pushing the plan not from the standpoint of seeking results right now but from a long-range view.

It can probably be said that these are the characteristics of the construction industry's research institute.

Research Organization Chart

Technical Headquarters
Hiroshi Kitamura, Chief

Bureau	Division	Office	Functions
Technical Planning Bureau, Tatsuya Nagai		Planning Office, Fumio Sato	-Planning and drafting of technical policy, drafting of research and development budget execution policy, promotion of pilot projects, technical committee secretariat
		Control Office, Shizuka Kudo	-General control of research study quality
		Patent Office, Taro Inoue	-Acquisition and control of industrial property, collection and control of patent information, coping with patent infringement cases, proposition screening committee secretariat
		Technical Information Office, Takeshi Hosokaya	-Research, coordination and settlement procedures concerning public announcement of new technology, promotion of intracompany exchange of technical information, technical information collection service
		Technical Contract Office, Tatsuya Nagai	-Advice and study on contract conclusion, cooperation in negotiations, control of contracts
Technical Development Bureau, Shiro Yajima	Division in Charge of Development, Akira Matsumura	Planning Technology Development Office, Akira Matsumura	-Intelligent building, high-rise RC collective housing, high-rise RC buildings, site planning, project planning
		Mechatronics Development Office, Shigeru Sakamoto	-Small aperture pneumatic transportation, external wall coating automation, tile peeling diagnosis device, various working robots, ice making, low-temperature technology
	Division in Charge of Development, Riichi Kobayashi	Underground Construction Methods Development Office, Hisanori Hotta	-High-rigidity foundations, mine landslide fortifying method
		Construction Methods System Development Office, Koji Yanagisawa	-Air film, frame film, large span structures, concrete purges, PC curtain wall
		Project Development Office, Joji Yamashita	-Permanent anchors
	Division in Charge of Development, Shu Isa	Foundation Work Development Office, Teiji Naito	-Foundation improvement, cofferdam construction, slope stabilization
		Civil Engineering Frontier Development Office, Tetsuya Hanamura	-Superconductive energy storage, underground LPG stockpile, geothermal energy and wave utilization systems, resin mortar, shield automation
		Ocean Development Office, Tadashi Matsumoto	-Petroleum platform, gathering place for fish, fish multiplication and cultivation, multicell ocean research
		Informationized Work Execution Development Office, Akito Suzuki	-Special surveying, tunnels, underground power plants, mine landslide fortification measuring, AI analysis

[Table continued on following page]

Division in Charge of Development Takeshi Taira	Space Development Office Takeshi Taira	-Space-related investigation
	Environment Development Office Yoichiro Umesu	-Waste final disposal, tissue culture, coping with NUTEC, SCR, electromagnetic wave shields, thermal ice, storage, thermal water storage, regional heat supply
	Information Media Development Office Hiroshi Matsutate	-MB application, tile peeling diagnosis device, pipe clogging diagnosis device, computer graphics, information communication advanced utilization
	Development Promotion Office Haruo Otsuka	-Theme search and investigation, development promotion, support of technology transfer

Technical Research Institute

Chief: Hiroshi Kitamura, Managing Director
Deputy Chiefs: Heiichiro Okawa, Eiichi Goto

Division in Charge of Planning Hiroshi Tsuruta	Planning Office Hiroshi Tsuruta	-Elaboration of research policy, operation policy, window business, publication and safekeeping of reports, collection of technical information data
Division in Charge of Control Masaru Ito	Control Office Tetsuro Watanabe	-General affairs, accountant's business, maintenance and control of facilities
	LA System Office Kazuhiro Enomoto	-Future plans, operational plans for electronic computer- and communication-related facilities owned by the Technical Research Institute, promotion of LAN
	Experiment Planning Office Toshio Yamada	-Advice, support for experiment plans, facilities and equipment acquisition plans, facilities and equipment maintenance, control and operation
	Chief Researcher Hiroshi Tase	-Dissemination and support of research results and technology
Division in Charge of Building Construction Tatsuo Mogami	Structural Material Laboratory Takashige Hattori	-Research on skeleton material aimed at building quality improvement and function strengthening
	Building Production Laboratory Kazuhisa Takeda	-Selection of building materials and construction methods, research on production technology aimed at quality assurance
	Structure Laboratory Seiji Yoshizaki	-Structural analysis of buildings, research on construction methods for upper sections and foundation structures
	Earthquake Proofing Laboratory Soichi Kawamura	-Estimate and evaluation of foundation and structure vibration produced by earthquakes, research on earthquake proofing and damping technology
	Soil Laboratory Yoshio Wakainochi	-Research on physical properties and evaluation of soil
	Chief Researcher Yasuhiko Tsuruta	-Dissemination of technology concerning facing methods

[Table continued on following page]

Division	Laboratory	Research
Division in Charge of Civil Engineering Eiichi Goto	Civil Engineering Materials Laboratory Takashi Naito	-Research on materials and structural characteristics, quality assurance, and production technology for civil engineering structures
	Foundation Laboratory Katsuhiro Nakura	-Research for enhancement of foundation-related production technology
	Rock Bed Laboratory Takashi Naito	-Research on physical properties and evaluation of bed rock
	Oceanography and Hydraulics Laboratory Yoshihiko Myogahara	-Research on basic and element technologies for practical use of water-area space
Division in Charge of Plan Motoaki Mukaino	Environmental Plan Laboratory Takeo Koike	-Research on amenities to improve living environments and the effective utilization of energy
	Acoustics and Psychology Laboratory Takeo Koike	-Research on improving quality of Acoustic performance of buildings, research on environmental psychology
	Disaster Prevention Planning Laboratory Kuniomi Suzuki	-Research on refuge, safety in times of disaster
Division in Charge of New Materials Yasuhiro Yamamoto	Material Chemistry Laboratory Hirokazu Hashimoto	-Research and development of new materials and research on improving basic physical properties of materials
	Material Development Laboratory Kuniyuki Tomatsuri	-Research on making new materials applicable to construction field
	Biotechnological Laboratory Seiji Kaneko	-Biotechnology research on efficient use of biomass
	Numerical Simulation Laboratory Heiichiro Okawa	-Research on understanding fluid phenomena through large-scale numerical analysis and visualization technology

What Taisei Corporation is now positively pushing is the development of the concept of engineering constructor (EC) and the business of "planning propositions." EC means not merely carrying out construction according to specifications but also conducting planning, projecting and designing of construction, and even equipment installation, and, further, it even includes maintenance. The planning proposition business, as its name indicates, makes it the central aim of the business to make propositions designed to reflect the intentions of the orderer.

Pursuing Research on Basic Technology That Is Also Applicable to the Construction Industry

"It is too late to start operations after the blueprints of a building or a civil engineering project are completed. We must start by making propositions in the first place," says Tatsuya Nagai, chief of the Technical Planning Bureau. He is in the Technical Headquarters of the head office where he is in charge of the planning of subjects for study.

How are the concepts of EC development and planning propositions linked with basic research on biotechnology?

"To what extent can the process of biotechnological research be understood from the standpoint of the constructor? It becomes a sales point for the construction industry. Let us take the case of constructing a food maker's research institute or plant as an example. It is no good to carry out design and construction merely by reading figures and following specifications. We must fully understand the flow of research or even the research of the other party itself, thereby discovering what the other party has in mind. After this, we can make a proposition to the other party" (Bureau Chief Nagai).

Not only that, but constructing the research institute itself has great significance.

"For one thing, we wanted to confirm our ability to create a research institute. In other words, it is a simulation. In fact, we made a research institute that is very interesting" (Bureau Chief Nagai).

It is believed that this sector came to be separated and transferred through the normal process of looking ahead. Says Deputy Chief Okawa: "When the decision was made to engage in full-scale biotechnology, we realized that the nature and tools of the work differed from those of the other laboratories. Thus it was concluded that it would be better to boldly separate this institute as a satellite institute rather than keep it in the same research institute."

Institute Chief Kaneko goes on to say: "There is an effort not only to push research but to read customers' words and aims and to translate them into the language of construction. To that end, it is required

This company appears to have a great deal of interest in ocean development. Not only in this institute, but in the Technical Research Institute as well, the Oceanography and Hydraulics Laboratory is doing well, and in the Technical Development Bureau, too, there is an Ocean Development Office, which handles more practical branches.

The company sometimes goes to the construction site on or in the sea, and conducts surveys, etc., often playing the leading role. The investigation sector, too, is making many achievements, such as developing an epoch-making system, but these will be discussed later.

Ryoji Okawara, who is in charge of plants, majored in agricultural chemistry in the postgraduate school of Tohoku University. He joined the company in 1986, when biotechnological research was started.

From Foundation to Systematization of Technology for Raising Seeds and Saplings

Aiming at the industrialization of agriculture, they handle issues ranging from the foundation to the systematization of technology for raising seeds and saplings.

Rice plants are also raised, but "we must know their original nature. Thus we are conducting this work for the purpose of study," (Institute Chief Kaneko) they say reservedly.

However, cell fusion and reproduction from protoplasts (naked cells without a cell wall) are also the subjects of research.

"To improve rice plants, the newest biotechnology is used. In a way, we have created a sanctuary where we can process the steps from seeds to saplings in artificial facilities. The creation of such facilities is, of course, in the domain of construction. Even though we cannot go so far as to attain the actual improvement of plants, it is important for us to know how to raise them in these facilities," says Technical Planning Bureau Chief Nagai.

There is a cultured plant growth environment control system called "multigreentron" [phonetic]. This can remove impurities by controlling temperature, humidity, luminous intensity, gas density, air current velocity, etc. This depends heavily on clean room technology. Research on clean rooms had been undertaken previously and applied to semiconductor laboratories, etc. It has been improved for use in biotechnological laboratories.

In the hothouse prepared especially for the institute, various species of foliage plants are being raised.

"These are not used for food, but if we have actually had this experience, we can speak convincingly when we come to give advice. In that sense, too, we are handling a wide range of plants" (Institute Chief Kaneko).

The biotechnological sector of Taisei Corporation is characterized by its lawn grasses. This technology is necessary for making golf courses. By researching the lawn grasses to be planted, those with a high level of weather resistance (species resistant to weather changes) and soil resistance can be efficiently produced.

"Since we have long been grappling with golf course construction, there are some who are strong in lawn grasses. Formerly, we used to choose a suitable strain from among the lawn grasses on the market. We will now be able to put the results of this research to our own practical use" (Bureau Chief Nagai).

Since Institute Chief Kaneko is reportedly the oldest in this institute, the other members must all be young. They are now in the stage of going to push their own work while studying to master the basic technology somehow or other.

Institute Chief Kaneko has it in mind to make this institute into an organization of about 50 members in the future.

As has been mentioned, the reorganization of the Technical Headquarters took place on 1 April. As part of this process, the Technical Research Institute was generally reorganized.

"The research system is very dynamic, so it is common for it to undergo changes" (Bureau Chief Nagai).

The Technical Headquarters includes the Technical Planning Bureau, the Technical Development Bureau and the Technical Research Institute.

The Technical Planning Bureau plans research. It determines the subjects for study by giving an ear to the needs of the work site and requests from other enterprises and the administration.

"We budget for as many as 150 to 200 research projects every year by grading them" (Bureau Chief Nagai).

There are some members who take charge of industrializing and acquiring rights to the achievements of research and development, and applying for the patents to them. It is also tasked with grasping and analyzing technical needs according to patent information.

It is also one of the bureau's responsibilities to decide in what way results obtained through joint development are to be commercialized.

They must push joint application for patents and the joint acquisition of rights.

Another important task for the bureau is to grant patent licenses for the achievements of third parties or, converseely, to determine the conditions for the introduction of the technologies of other companies (including those from foreign countries).

Technical Research Institute To Grapple With Long-Range and Basic Research

The Technical Development Bureau is closely connected with the work-site operations sector of the head office and seeks short-term results. By contrast, the Technical Research Institute is grappling with longer-term and more basic research. However, when laboratory results come out and their practical application is thought to be near, for instance, they sometimes are transferred directly to the development bureau.

The Technical Research Institute started functioning in June 1960 as the Toyosu Research Institute at Toyosu, Koto Ward, Tokyo. It was relocated to the present site in August 1979.

The company's managing director, Hiroshi Kitamura, serves as the institute's chief, and two persons, Heiichiro Okawa and Eiichi Goto, serve as deputy chiefs.

Its research areas can roughly be divided into building, civil engineering, planning and new materials. The last new materials system was transferred from the Technical Development Bureau to the Technical Research Institute in the April reorganization. It has two laboratories--the Material Chemistry Laboratory and the Material Development Laboratory.

"Most of the structures use natural inorganic materials. Naturally, however, there are also some functions they lack. Thus it is necessary to make up for them by using new materials" (Bureau Chief Nagai).

The Technical Research Institute underwent a big change in the most recent reorganization. The Technical Development Office was divided to create two new development offices.

One of these is the Informationized Work Execution Development Office, which, unlike the new materials system, was transferred from the Technical Research Institute.

One office that can claim to be in the van of the new era is the Space Development Office. In a way this is a development looking ahead to the future, and it can possibly be said to represent the dream of the construction industry.

"A desire to expand the space of human movement is our innate will, and also meets one of the big needs of society. Following the oceans and underground is space" (Bureau Chief Nagai).

One immediate task is to make a weightless state facility. Falling from a high altitude produces a state of weightlessness, and experiments designed to make the most of its characteristics can be conducted. In addition, construction knowhow--on a base for ground-launched rockets for instance--can be acquired.

"If possible, we want to introduce construction on the lunar surface into our repertoire" (Bureau Chief Nagai).

One entirely new establishment there is the Numerical Simulation Laboratory. The laboratory chief is Okawa, who is also deputy chief of the Technical Research Institute.

"A newly emerging field of study is metrical fluid engineering or metrical engineering. This is the study of what has been achieved experimentally or theoretically by using a supercomputer" (Technical Research Institute Deputy Chief Okawa).

Okawa joined the company in 1960 after graduating from the architectural engineering program of the Musashi Institute of Technology. He majored in sonics.

"When this company builds a hotel, I invariably stay at it as a matter of interest. In the case of a concert hall, for instance, I will go to listen to a performance there even if it was constructed by another company. First, I listen to it as the sound of the hall without giving consideration to the conductor or the orchestra. As it is my way to enjoy it as music only after listening to it several times, I will visit one hall a good many times. And, if I give no thought to how I would build it should I be its constructor, it is of no use. I have only one thought in constructing a hall: to build it so that one's whole body is enveloped in music. I wish we could create a space where both performers and their audience could unite in a single body and yet be fascinated by the music" (Okawa).

By incorporating "psychology," the sonics sector has become the Acoustics and Psychology Laboratory. Psychology means studying the room environment not in terms of its physical properties, such as temperature and humidity, but from the standpoint of human habitation. This perspective is unique to Taisei Corporation. This laboratory is conducting so-called interdisciplinary research on the border between architectonic and psychology. It currently has three researchers who learned a bit of psychology from an architectural perspective.

Assigning Environmental Psychology Sector to Independent Laboratory Also Planned

"Environmental psychology, for instance, is not something originally conducted by the construction industry. However, our company is making the space in which man is to live. We need to show our customers the way to make a good environment at the same time show them the cost of doing so. We should make every effort to do this."

Okawa goes on to talk about the concrete details:

"It is not merely color or temperature that we consider. We go so far as to study the effect a certain combination of color and temperature would produce. When considering a high-rise housing project, we find that the window size, for instance, has a great influence. Looking at a building from the standpoint of those who will live in or use it, we can efficiently use our findings in its design."

This research institute has a plan to develop this environmental psychology sector further into an independent laboratory.

Only in Japan is there a construction company that has such a research institute, and thus it is a wonder to overseas interests.

"In foreign countries, the construction industry carries out construction only as specifications prescribe. Research organs, for concrete or anything else, are all state-operated. In Japan, we conduct design and construction by taking into account the customers' needs. Research organs for that purpose are increasing rapidly. If we expand them to include those for biotechnology and psychology, they may be more and more surprised" (Kaneko).

"There are voices, both inside and outside the company, asking why Taisei conducts basic research that does not pay, like biotechnology or psychology. In this respect, the top echelons of this company are understanding, and we are operating rather freely" (Okawa).

The following are examples of how our research results are put to practical use at the actual construction site.

The construction industry is not such that, having a sample of the real thing or a model at the time it accepts an order, it erects a structure as shown and sells it. Although structures differ from each other, each of them incorporates technologies resulting from research and development. A look at a building reveals that it contains conventional technologies, new technologies, and also technologies developed jointly with allied industries (so-called subcontractors). And, further, it involves technologies the construction industry has developed by itself.

For example, the construction of liquefied natural gas (LNG) tanks involved many difficulties. First, because LNG must be stored at a low temperature (-173°C), its surroundings are affected. There is also the problem of constructing a large structure on a soft piece of reclaimed land.

In this situation, the continuous underground wall construction method is used. The standard depth is 50-60 meters, but recently some units have been built at a depth of 150 meters.

As a demonstration experiment for theoretical analysis and projection, a continuous wall was constructed by digging a 160-meter hole in a piece of reclaimed land in Kawasaki. Its shape is close to a square 2.5 meters thick and 3 meters wide. High technology also plays a part in digging straight. It is a technology to measure accuracy by using laser beams. This measuring instrument was used jointly by three construction companies, the machine maker and this company. This continuous underground wall technology is the product of research and development based on the Tokyo Bay transverse road project.

On 10 April of this year, the Kojima-Sakaide route of the Seto Ohashi bridge was opened to traffic. Other routes are also under construction. This bridge construction also incorporates many technologies.

Prior to the actual construction work, a survey was conducted. A device called the "underwater civil engineering work control sonar" attracted attention as an ocean current and sea bottom investigation system. Measurement by sending supersonic waves from a ship has been conducted before, but it has now become possible to let a computer store the data and draw a figure automatically. Further, it is possible to grasp the topography three-dimensionally and even to draw it as a solid figure. This is what was developed by the Ocean Development Office of the Technical Development Bureau. It was highly evaluated in the experiment at the work site in the Straits of Akashi.

Blasting operations are sometimes carried out to facilitate digging on the sea bottom. This is done to expose the hard bedrock so the bridge pier can be built on it. To estimate and analyze the extent of sea pollution that results from such an operation, an experimental water tank is used. There is also the problem of the fish population. To conduct blasting operations without killing the fish, a technology that generates sonic waves that the fish dislike was used in advance to drive them away.

'Heat Recovery System Using Wave Power' for Wave-Activated Power Generation

In an effort to prevent a misfire, radio-operated detonation was employed for the first time.

There is also the problem of maintenance after the bridge is completed. The physical properties of the bridge are always measured. As a matter of course, the traffic volume is also automatically measured for use in traffic control.

Not only is the firm involved in this sort of actual work, but it sometimes has to grapple with plants for a demonstration experiment. One of these, the "heat recovery system using wave power," is discussed below.

Due to the oil shortage in 1973, both the government and private industry began to search for alternate energy sources. Even if other resources are found, they are not inexhaustible. Thus, measures were taken to grapple with natural energy. Solar light, wind and geothermal heat, for instance, are available inexhaustibly without paying any price.

Taisei Corporation began to explore wave-activated power generation. This is probably the most desirable option for practical use in Japan, an island country.

Having been granted an alternate energy development subsidy by MITI's Agency for Industrial Science and Technology, in 1985 this company finally started a plant construction project as a "demonstration experiment of the heat recovery system using wave power."

The company organized a special staff. It comprised some 10 members drawn from the Civil Engineering Frontier Development Office, the Technical Development Bureau and also from the Technical Research Institute, the Civil Engineering Design Division, the Civil Engineering Division, etc.

The first step was to find an installation site. Not just any place along the seashore will do. Three requirements must be met. First, the waves there must be high. Second, there must be waves throughout the year. Third, the project must be accepted by the fishermen's cooperative association and the local population of the area concerned.

From among several suitable sites, Neya fishing port of Yamakita Town, Iwafune, Niigata Prefecture, was chosen.

The technology that served as the base for this project was that used in the water tank experiments conducted at the Technical Research Institute beginning in 1979. This institute has two water tanks. One is a two-dimensional water tank 45 meters long, 80 meters wide and 1.6 meters deep. The other is a three-dimensional water tank 35.5 meters long, 17 meters wide and 1.6 meters deep.

These water tanks were used in an experiment on wave-power energy recovery using a small model. The basic data acquired were finally put

to practical use in the plant for the demonstration experiment using actual waves.

In late June 1986, the installation of a box-shaped unit was conducted, but it was under unfavorable conditions just after a typhoon. This unit was a reinforced concrete structure 13.3 meters wide, 14.5 meters high and 7 meters long. It weighed 1,300 tons.

It was towed by a boat from Iwafune Port about 40 km away from the site. Work began at 0230 hours and was completed at 1100 hours.

In December, the system started operating. The collected energy is converted to thermal energy and is sent out as hot water.

Given waves 2 meters high, hot water at a temperature of 45°C can be made at a rate of 1 m^3 per hour. This hot water can be used for such purposes as household heating and hot-water supply, hothouse horticulture, fish farming and snow melting.

Researchers' Strength Is Their Ability To Generate Ideas Themselves

Briefly, the principle of this system is as follows: There is a concrete vessel with an air room designed so that the seawater goes in and out. When the waves roll in, the water level in the vessel rises, and the air therein is compressed. The air current thus created turns a turbine. This turbine is called a Wells [phonetic] turbine, which is structured so that it always revolves in a fixed direction even in a reciprocating current. The rotatory power of the turbine is directly converted to heat by an improved version of the "eddy-current dynamometer," and the cooling water from outside is recovered as hot water.

The field office carried out a computer analysis of the data. It is connected to the plant by an optical fiber over which the data are being transmitted.

Tetsuya Hanamura, chief of the Civil Engineering Frontier Development Office, talks about this system: "This system, which can generate 40 watts of heat on a regular basis by producing 40°C hot water, has a drawback from the standpoint of cost now that the oil situation has improved. But looking toward the future, it is expected to be put to practical use for the utilization of hot water for the purpose of activation of the area concerned and at the same time as a breakwater having wave killing functions."

Even if the practical use of wave power is still a matter for the future, the extent of this company's attention to the marine sector can be understood.

Every year when new employees come in, they receive training in groups on the building construction system, the civil engineering system, etc. The research institute members are no exception to this practice.

"If they joined the company purely for research, they may have no knowledge about the construction industry. However, I think it is bad for them to have no knowledge of what their company is doing. It is necessary for them to experience work-site operations for about half a year."

Okawa goes on to talk about the qualities he looks for in researchers.

"They must be men who can consider things for themselves. In looking at our most recent researchers, they are excellent with regard to problems where they know where to find the answers. On the other hand, they are very poor at dealing with problems where it is unknown if answers can be given or not. This is too bad."

Institute Chief Kaneko refers to the researcher he regards as ideal as a "researcher with an ear for listening."

"The construction industry must read its customers' minds. It is necessary to externalize the image of the other party, that is, what type of building the other party wants to construct. The researcher with an ear for listening is also what I am aiming at."

As if to supplement Kaneko's idea, Okawa gave a concrete example:

"He means that in constructing, say, a music hall, it is desirable that its designer be a man who not only considers the floor space or capacity per head or how long the reverberation time lasts, but who also really understands the heart of music. In ocean development, too, they must be such researchers who will not only observe the legal standards but will also give consideration even to the living things therein. This means that they will go to great lengths to consider if there really will be no influence even 10 or 20 years from now. That is Kaneko's idea. Young members of his institute are working with an understanding of his idea."

Following the new institute in Chiba, an experiment building for the planning system was newly established in June in the compound of the Technical Research Institute. For this building, a newly developed earthquake-free construction method has been adopted.

This building is a four-storied edifice with a gross weight of 2,200 tons. The earthquake-free structure is designed with eight supports that wear synthetic resin "shoes" and slip on a stainless plate, thereby resulting in the absorption of a tremor.

It is feared that if computers should be disabled in a big earthquake, an information panic would occur and many industries would stop operating. Should the effect of this new earthquake-free construction method be proven, there would be a great demand for it as something that is serviceable for intelligent buildings.

20150/09599

Fine Ceramics Process Technology

Ultrasonic Process Technology

43064051 Tokyo KIKAI TO KOGU in Japanese May 88 pp 49-59

[Article by Koji Nirei, Nippon Denshi Kogyo Co., Ltd.: "Processing of Ceramics by Ultrasonic Processing Machine"]

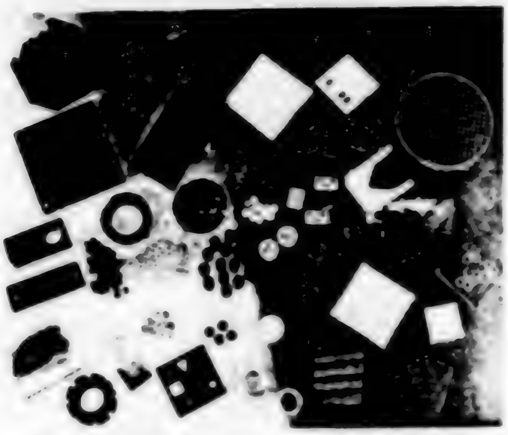
[Text] 1. **Introduction**

The ultrasonic processing method extends ultrasonic vibration to a processing tool by pushing it against a workpiece through a mixed slurry of water and grinding particles, and initiates impact crushing by shooting the grinding particles into the workpiece.

Although the amount of processing achieved by one shot of the grinding particles is small, a practical processing speed can be obtained since impact crushing is repeated tens of thousands of times per second. The impact crushing processing method is appropriate for processing hard and brittle materials, i.e., the materials showing a small "tensile strength anticompressible strength," such as ceramics. This method is widely utilized.

Since the amount of material crushed per impact by the grinding particles is very small in this processing method, adequate processing accuracy can be obtained with relative ease, and the smoothness of the processed surface is good. Large distortions are not seen on the workpieces. The miniaturization of the processing dimensions and the improvement in accuracy are noted as current trends of this method.

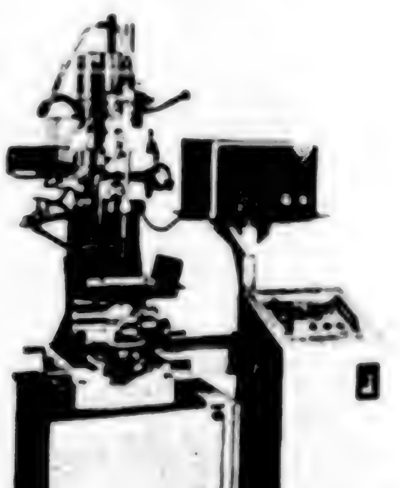
On the other hand, due to the advent of a large workpiece composed of fine ceramics, there is the tendency for a processing machine to have an exclusive use, thereby fractionalizing its operating method. When the amplitude of a processing tool is looked at, its application ranges from small amplitudes of several μm to maximum amplitudes within the limits of the tool's strength. Photograph 1 shows several kinds of processed products.



Photograph 1. Examples of Processed Products (Materials: graphite, ferrite, silicon nitride, alumina, superhard alloy, quartz glass)

2. Ultrasonic Processing Method and Characteristics

Photograph 2 and Figure 1 outline the processing machine and method. The vibration of the vibrator driven by the oscillator is longitudinal, i.e., expansion and contraction occur along the direction of the axis. Because the magnitude of this vibration itself is too small, its amplitude is magnified by the cone and the horn designed and made to correspond to the equivalent half wavelength. The amplitude of the tool attached to the top of the horn can reach 15-50 μm, and the frequency is in the range of 15-30 kHz. The vibrator, cone, horn, and tool are concatenated. Their composition becomes the vibration system, resonating at the resonance frequency of the vibrator.



Photograph 2. Ultrasonic Processing Machine Model UM-1000 EA (Output: 1 kW; frequency: 16 kHz, nickel vibrator, water cooling)

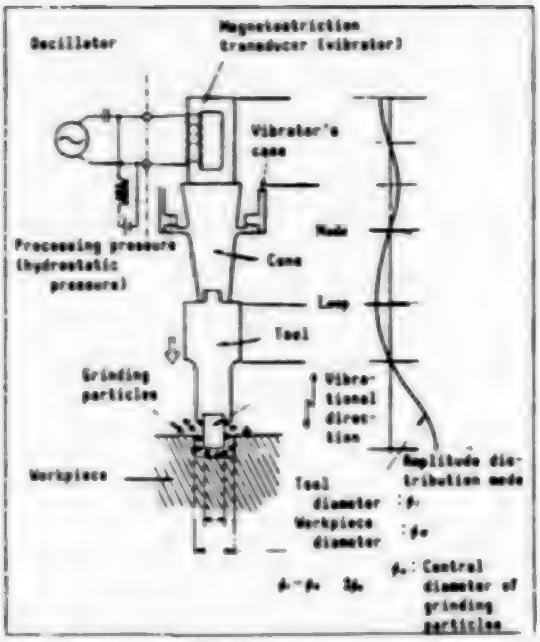


Figure 1. Processing Principle

The grinding particles composed of B_4C and SiC are mixed with water, becoming a slurry. This slurry is poured onto the processing surface, and the tool is pushed against the processing surface. The grinding particles migrating on the processing surface are shot into the processing surface by the tool's vibration, and a small amount of the workpiece is gradually crushed. The shape corresponding to the edge shape of the tool is copied and cut into the workpiece.

A processing clearance occurs that corresponds to the average diameter of the grinding particles between the tool and the workpiece. Therefore, the diameter of the tool should be twice as large as the diameter of the grinding particles (normally, the grinding particles have an average diameter of 20-50 μm). The water suspending the grinding particles carries and discharges the grinding particles and waste materials.

Mild steel, stainless steel, and spring steel are employed as the tool materials. They are mechanically attached to the tip of the horn by soldering or brazing. When a small tool with a small amplitude is used, screwing or jamming can be practically applicable. Since this method uses the grinding particles, tool abrasion is inevitable. Accordingly, the tool should be recognized as being expendable.

Table 1 shows processing characteristics classified by material. The processing cutting speed varies with the amplitude of the tool, the compressible strength of the grinding particles, the diameter of the grinding particles, etc. The processing properties of ceramics vary with the composition and manufacturing processes, even if the same brand of ceramics is used. This means that some exceptions exist. The

Processing Characteristics Classified by Materials

Materials	Grinding particles	Processing speed (mm/min)	Processing ratio	Processing pressure (g/mm)
Glass	SiC, #320	6.0	150-250	30-50
Barium titanate	SiC, #320	6.5	70-110	100
Ferrite	SiC, #320	6.5	90-130	80-150
Porcelain	SiC, #320	6.5	200-240	30-50
Quartz	SiC, #320	5.5	100-140	80-150
Graphite	SiC, #320	8.4	200-300	230
Silicon single crystal	SiC, #320	3.5	200	230
Ruby	SiC, #320	0.6	8-12	
Ruby	B_4C, #280	0.8	5-7	
Alumina	SiC, #320	3.0	30-40	290
Alumina	B_4C, #280	3.6	20-25	290
Superhard alloy G-2	B_4C, #280	0.2	2.0	330
Superhard alloy S-type	B_4C, #280	0.3	4.0	330
Silicon nitride (HP)	B_4C, #280	0.6	2.0	
Compax (artificial diamond sintering body)	B_4C, #280	0.17	0.014	
Silicon carbide (HP)	B_4C, #280	0.7	2.3	
Zirconia	B_4C, #280	0.4	1.3	
S55C	B_4C, #280	0.4	1.4	400
S55C hardening (H_RC46)	B_4C, #280	0.26	1.0	150-200
SKH3	B_4C, #280	0.4	1.5	250-280
SKH3 (H_RC60)	B_4C, #280	0.26	0.7	350
SKD6	B_4C, #280	0.3	1.1	200-250
SKD6 (H_RC46)	B_4C, #280	0.2	0.9	250-300
SK5	B_4C, #280	0.3	0.8	200-350
SK5 (H_RC60)	B_4C, #280	0.36	1.6	450
SUS304	B_4C, #280	0.3	1.5	250-280

(Processing conditions) Output: 150 W; Resonance frequency: 16 kHz; ϕ3mm solid circular rod (piano wire)

(Grinding particles) SiC,#320: silicon carbide average diameter of 48 μm

B_4C, #280: boron carbide average diameter of 40 μm

Processing ratio = processing depth/abrasion of a tool

characteristic values listed in Table 1 depend upon the priority given to the processing speed. Practically speaking, in order to avoid the occurrence of cracks or to ensure accuracy, smaller values than those given are used in some cases.

From the processing characteristics of metallic materials, it is seen that the metal mold steel SKD6 has the advantage of less abrasion. Due to the diffusion of the wire discharge processing method, a cutting tool is not always necessary for tool formation. In some cases, this method is economical.

This processing method can be applicable to hard and brittle materials, and does not depend upon the electrical condition of the workpiece. As examples of materials which can be processed by this method, glass, quartz glass, new glass, semiconductor elements, ferrite, graphite, various kinds of ceramics, precious stones, and diamond are noted (Photograph 3 [not reproduced]).

Since the processing method is an impact crushing processing method in which slight vibration along the direction of the tool axis occurs, the rotation of the tool and the relative motion between the tool and workpiece are generally not needed. Therefore, this method can be applied widely to such processing tasks as punching (circular holes and other hole shapes), spot facing, clipping, cutting, grooving, screwing, engraving, and lapping.

In addition, as an example that makes the most of the characteristics of this method, simultaneous processing of multiple items employing multiple tools should be mentioned. For example, hundreds of holes the diameters 0.5 - 2 mm could be simultaneously processed with a uniform pitch on a square alumina board, 50 mm x 50 mm, or more than 500 elements, 1.8 mm in diameter, could be punched out simultaneously from a silicon wafer with a diameter of 50 mm.

The processing surface roughness is almost proportional to the diameters of the grinding particles used. In the case of glass, which can be processed easily, it becomes 2 to 8 percent of the central diameter of the grinding particles. On the other hand, in the case of ultrahard alloys and silicon nitride, which are difficult to process, it becomes 0.8 - 2 percent. This method can be utilized to give high efficiency to grinding fine holes, such as a diamond die for line drawing. At the same time, it is also used for grinding metal molds and modifying dimensional variations by a hardening process.

3. Processing Machine

The processing machine is composed of the vibrator and pressurizing device. They consist of the mechanical portion, which supports the workpiece and maintains the positional relationship between the tool and the workpiece with good precision, and the vibrator/grinding particle circulation system. A description of these components will be discussed.

3.1 Vibrator System

This is the portion which converts high-frequency electric power supplied from the oscillator into mechanical vibration, i.e., ultrasonic vibration. The electricity-sound conversion element, i.e., the vibrator, is made from laminate nickel thin plates or ferrite materials (sinter) of the magnetostrictive type. In addition, the Langevin-type vibrator is used for

electrostrictive purposes. For a general-purpose machine, the vibrator composed of nickel is very easy to handle currently.

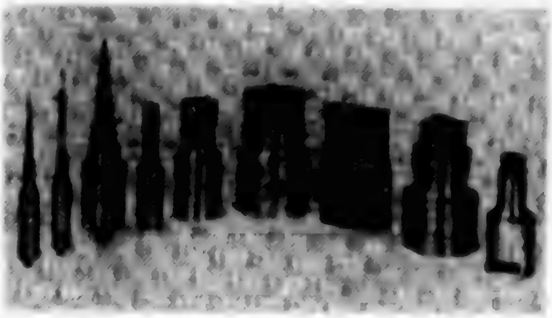
For instance, when the length and dimensions of the tool differ from those of the original design and, consequently, the resonant frequency of the horn portion is slightly different from that of the vibrator, a detailed recalculation of the resonant length of the horn or velocity vibration ratio (amplitude magnification ratio) is not required on each occasion since the degradation of processing characteristics is slow in this method.

Even in the case of overloading, i.e., an excessive tool or large processing area is encountered, it is still usable in many cases if the declination of the processing speed is within certain limits. This means that the advantage of the ultrasonic processing method, which facilitates the shape modification of a workpiece not only at the prototype stage, but also at the mass production stage, could be heightened further.

The cone is attached to one end of the vibrator, and can magnify and transmit the amplitude of vibration generated in the vibrator. Since the amount of heat generated by the nickel vibrator is large, it is fixed by the water cooling case which supports the mechanical vibration system, i.e., the vibrator, at the vibration node site in the cone.

At the output terminal of the cone, a screw is provided to which a horn is attached. The vibration system concatenated with the vibrator, the horn, and the cone resonates under the resonance frequency of the vibrator as one body. The horn, including the processing tool attached to its tip, is designed so that its resonant length becomes multiplied by a half wavelength under the resonant frequency of the vibrator.

The horn possesses the metamorphic function by which the vibration amplitude of the thin edge side becomes larger than that of the thick edge side. As seen in the transformer of an electric circuit, it conducts the metamorphic action for force and speed in the mechanical vibration system. The practical example of the horn is shown in Photograph 6. References 1-4 [not reproduced] can be referred to for details.



Photograph 6. Practical Examples of the Horn

Various shapes are available for the most commonly used horn. They include, for instance, exponential and conical shapes, whose cross sections become smaller approaching the tip, or a step shape where bars with different sizes

of uniform cross sections are connected. Depending upon the purpose, these shapes are used with modifications or in combination. materials for use in the horn include carbon steel, which is commonly used for mechanical structured application, and stainless steel.

The processing capability of the device is restricted by the allowable input and applicable frequencies of the vibrator. The establishment of a processing area, the shape and size of the tool, and velocity variation ratio of the horn that match the processing capability of the device can be estimated with relative ease (Figure 2).

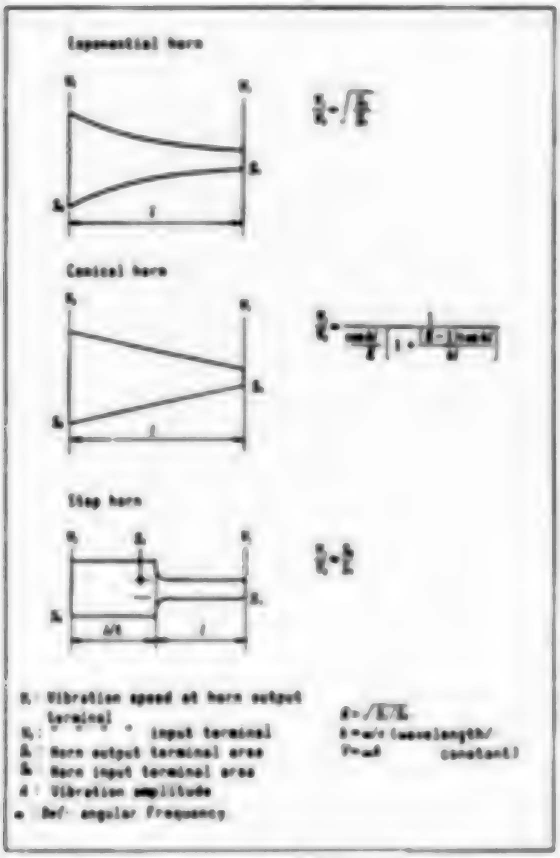


Figure 2. Horn Shape and Velocity Vibration Ratio

However, the selection of the appropriate cone shape is very important from the aspects of maintaining processing precision, residual cracks on processed surfaces, and reducing chipping.

Regarding tool vibration, the existence of only a longitudinal vibration component parallel to the tool axis is the ideal situation. However, when the tool is attached to the tip of the horn, the possibility exists that bending can be caused due to the buckling during processing, as well as lateral and torsional vibrations. In most cases, these are not desirable. These phenomena may cause the precision to deteriorate, chipping to occur, and the processing speed to decrease.

The selection of the horn shape involves choosing the appropriate shape corresponding to the dimensions of the tool and required processing conditions, and to pay attention to problems associated with vibrations when the horn is combined with the tool.

The vibrational characteristic of the tool is believed to be one of the most important factors when the fine processing of anisotropic materials, including various kinds of single crystals, takes place or when residual cracks on processed surfaces become a serious problem.

Generally speaking, judging from the above viewpoint, the exponential shape produces better results than the easily-manufactured step shape or conical shape. A selection process to obtain a high Q (resonance intensity) seems to be preferable.

Regarding the horn shape and processing characteristics, emphasis has been placed on pursuing the processing speed and vibration system efficiency. Along with the diversification of processing demands and the improvement of processing quality, data involving the vibrational characteristics of the horn and processing characteristics are thought to be necessary from the viewpoint of the shape of the horn tool, processed surface properties, and processing precision.

3.2 Pressurization Mechanism

The pressurization mechanism is the device within a guide function that accurately sends a workpiece in the direction parallel to the direction of the tool axis (Figure 3). A small device usually adopts the oil pressure method that pushes a workpiece up and presses it against the tool. In the case of a large device , the balance method in which the vibration is suspended at one end of a lever and the tool is pressed against the workpiece is commonly adopted. In either case, the structure is intended to reduce friction at the movable portion as much as possible, to achieve a sufficient feeding sensitivity (for the case of the balance method, the quantity of "the inclination of a balance/weight" increases), and to assure an effective braking function. Otherwise, the workpiece may fracture and deformation of the tool may occur.

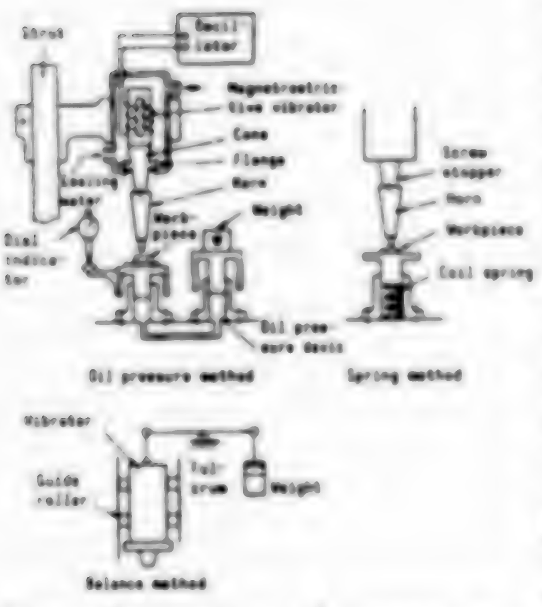


Figure 3. Outline of Pressurization Mechanism

In addition, whether the feeding sensitivity is good or bad is one of the factors governing the success and limitations of fine processing. It also influences the amount of hair cracks on processed surfaces.

For example, when processing materials for electronic parts, past experience has actually indicated that the feeding sensitivity exerted a great influence on the electric characteristics and yield.

The establishment of processing pressure is indicated by the figures listed in Table 1, and processing takes place with some vibration in the processing pressure. On the other hand, from information obtained by the processing depth indicator during processing, it is easy to establish appropriate conditions. Its limits are relatively broad, as seen in Figures 4 and 5. This feature is one of the advantages offered by the nickel vibrator, which can be handled easily. The processing pressure per unit area becomes smaller as the processing area becomes larger.

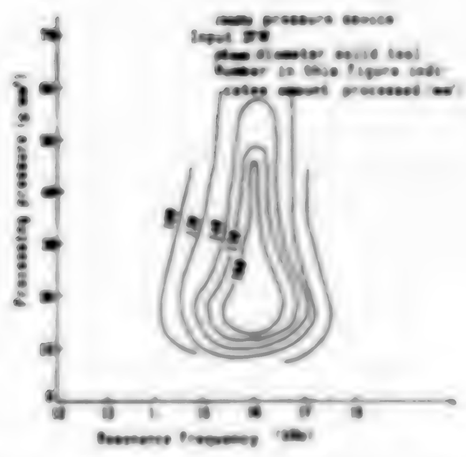


Figure 4. Horn Resonance Frequency and Processing Characteristics

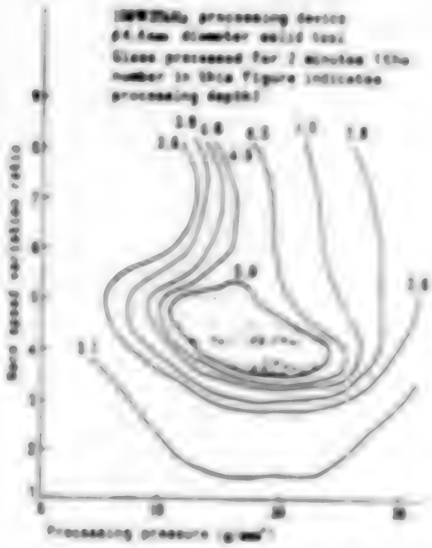


Figure 5. Speed Variation Ratio, Processing Pressure, and Processing Speed

When the depth of the processing cutting increases, the circulation of the grinding particles worsens, and the processing speed decreases. Therefore, in order to release the tool from the workpiece and to replace the old grinding particles with new ones, a device should be able to fluctuate the tool or the workpiece over a constant time interval on the pressurization mechanism.

3.3 Oscillator

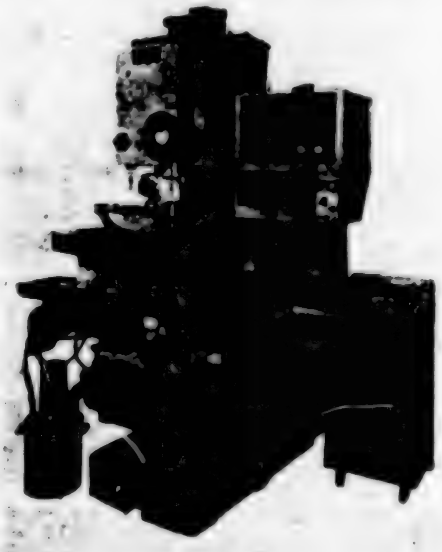
The oscillator supplies high frequency electric power to the vibrator synchronized with the resonant frequency of the vibrator. Its circuit composition consists of a manual control oscillation circuit that controls

the oscillation frequency by manual dialing, a main oscillation electric power amplification circuit, and an automatic frequency chasing oscillation circuit, since the resonant frequency of mechanical vibration systems, such as the vibrator, fluctuates during operation due to the fluctuations of the load and its heat generation. When a discrepancy exists between the driving frequency from the oscillator and the resonant frequency of the vibrator, the amplitude of the vibrator decreases and efficiency worsens. Therefore, the circuit is designed so that the vibration of the vibrator is electrically detected, the circuit that oscillates with that frequency is provided, the driving frequency from the oscillator automatically matches the resonant frequency of the vibrator, and the mechanical vibration system is able to maintain its best vibrational conditions.

The applicable frequency of a high power general-purpose device is about 16 kHz, corresponding to the resonant frequency. This device is easy to use, and is commonly utilized.

A 25 kHz device that has a large vibrational speed with a small amplitude is effective for processing a hole with a small diameter or a very fine hole. Therefore, it is mostly an exclusive device with little power.

As an example of a processing machine with a large output, Model UM-1500SA made by Nippon Denshi Kogyo is shown in Photograph 7.



Photograph 7. Ultrasonic Processing Machine, Model UM-1500SA

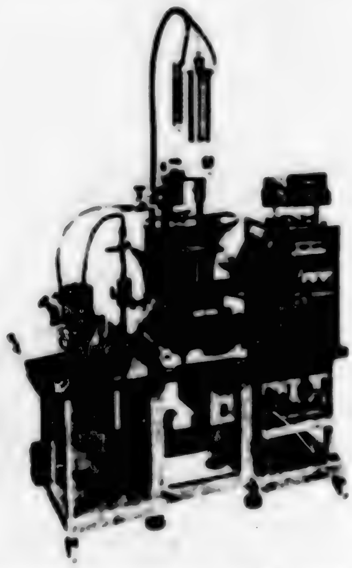
This machine is designed so that all processes, except the one to set up the workpiece, are automated, and an operator to watch the machine when processing fine ceramics or a large workpiece that requires a relatively long processing time is not necessarily required.

The vibrator installed in this machine is the nickel magnetostrictive type with a frequency of 16 kHz. The processing pressure is applied by dead weight after the dead load of the vibrator's head is equilibrated by a

balance. The maximum dimensions that can be processed by this machine are ϕ75 mm for a solid circular bar, □55 mm for a solid square shaped bar, ϕ100 mm for a hollow circular bar, □70 mm for a hollow square shaped bar, and a groove with a width of 22 mm and a length of 120 mm.

In addition to the large machine described above, Nippon Denshi Kogyo Co., Ltd., also manufactures and sells other processing machines, including small machines such as a precision fine hole processing device with an output of about 10 W.

Another example is Model UM-500DA-P, also made by Nippon Denshi Kogyo (Photograph 8). This machine provides the grinding particle absorption device.



Photograph 8. Ultrasonic Processing Machine, Model UM-500DA-P

Under this processing method, the grinding particles are crushed and become smaller during processing. Accordingly, new grinding particles must constantly be supplied on the processing surfaces. When the cutting depth is shallow, processing proceeds linearly, proportional to the processing time. However, as the cutting depth deepens, the processing speed decreases since the circulation if the grinding particles worsens.

When processing with a deep cutting depth, the adoption of a fluctuation mechanism to separate the tool or the workpiece previously mentioned toward the direction of the tool axis and to insert new grinding particles may be needed. For the case of the hollow tool, as seen from the deep hole processing characteristics shown in Figure 6, the method by which the grinding particles are absorbed through the hollow portion of the tube is very effective.

This machine is a middle-sized processing machine that equips the nickel magnetostrictive vibrator with an input of 500 W and frequency of 16 kHz, and an oil pressure-type pressurization mechanism for processing feeding of 50 mm.

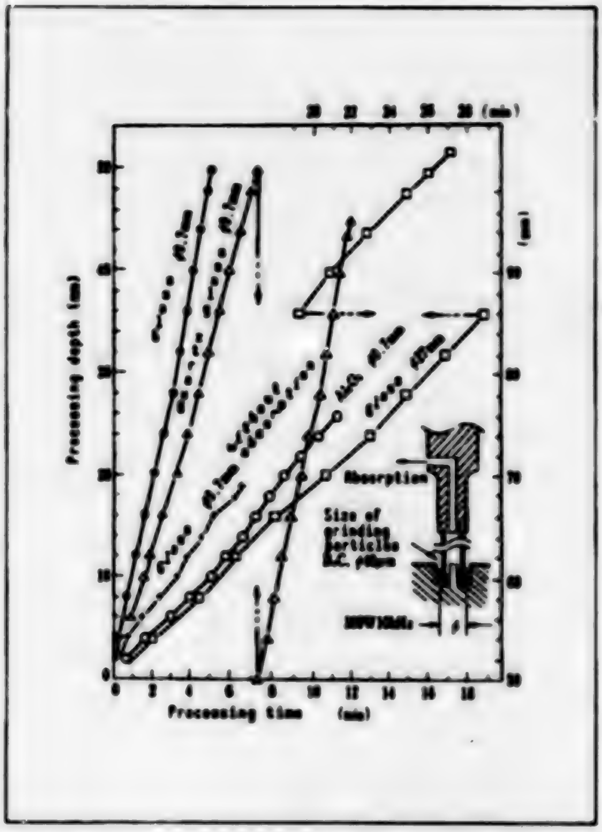


Figure 6. Deep Hole Processing Characteristics (Absorption)

The dimensional limits processed by this machine are ϕ0.4 - 40 mm for a solid circular bar, □0.3 - 28 mm for a solid square shaped bar, ϕ0.4 - 60 mm for a hollow circular bar, □0.3 - 44 mm for a hollow square bar, and a groove with a width of 0.4 - 10 mm and a length of 100 mm. When the hollow tool is larger than ϕ1.5 mm, absorbing processing is possible. In order to absorb the grinding particles, negative pressure is generated in the piping of the grinding particle circulation system. A portion of the grinding particles poured into the processing surfaces through negative pressure are absorbed through the hollow holes of the tool and the horn, and are returned to the grinding particle reserve tank. In this system, an additional pump to absorb the grinding particles and complicated piping is not necessary, but instead, a rather simple system has been adopted. Therefore, when exchanging the sizes of the grinding particles, only the tank and pump having different grinding particle sizes are replaced.

The effectiveness of absorption becomes apparent when the cutting depth is more than 4 mm for glass, and more than 1 - 2 mm for fine ceramics that require a relatively longer processing time.

4. Processing Speed and Precision

In this processing method, the following factors influence the processing speed and accuracy:

(1) Applicable frequency
(2) Amplitude of the tool
(3) Processing pressure
(4) Material and size of grinding particles
(5) Grinding particle supply method

Detailed functions and mechanisms of the processing characteristics mentioned above can be found in References 2 and 3 [not reproduced]. In this article, a discussion is presented showing how these factors relate to the processing speed and precision, which are usually incompatible with this processing method, and exert an influence on processing results.

To show the relationship between the processing speed and precision, the general relationship among the characteristic factors is represented by Figure 7. In order to increase the processing speed, a large amplitude and high processing pressure should be chosen, and large grinding particles with high fracture strength should be used.

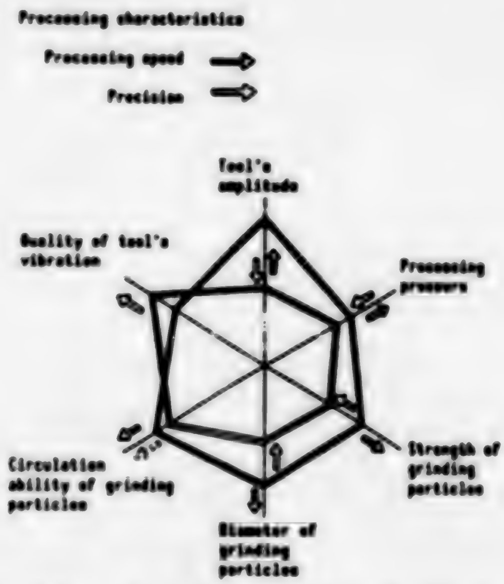


Figure 7. Relationships Among Processing Characteristic Factors

On the other hand, from the viewpoint of processing precision, selecting a small tool's amplitude and processing pressure and small grinding particles with relatively low strength brings better results. For this case, in the relationship among the characteristic factors, appropriate values correspond to each workpiece. Only the drastic change of one factor will lead to poor results.

If the amplitude of the tool is increased, the processing speed is improved. The processing speed is almost linearly related to the amplitude of the tool. Its practical limit is about 100 μm (peak to peak amplitude) due to restrictions involving the tool and horn strength. For fine processing, in order to avoid the fracture and chipping of the workpiece and to assure precision, a small amplitude of several μm must be maintained in some cases.

Regarding the properties and size of the grinding particles, when the processing speed is believed to be important, B_4C particles with large diameters, which have highest strength of the grinding particles generally used, are appropriate. However, since grinding particles larger than the amplitude of the tool cannot reach the processing surfaces, a suitable size corresponding to the amplitude of the tool is found.

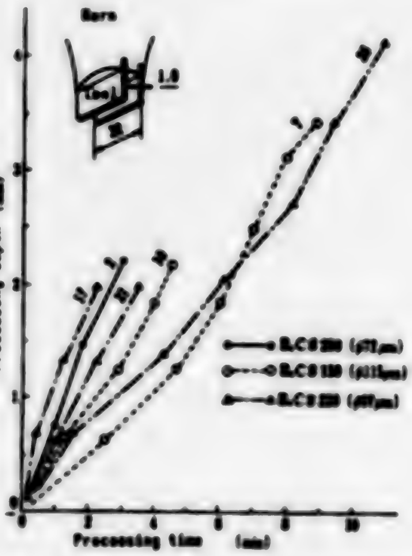


Figure 8. Processing Characteristics (I) of Si_3N_4(HP)

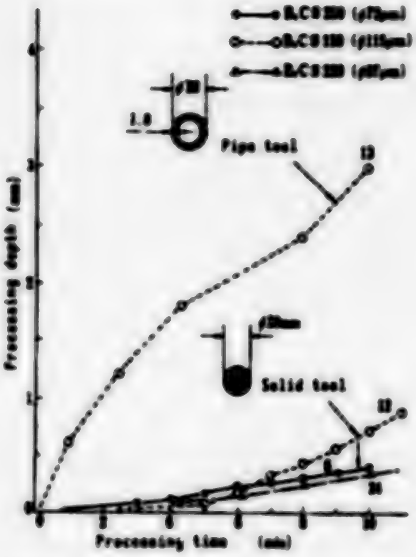


Figure 9. Processing Characteristics (II) of Si_3N_4(HP)

In addition, as seen from the processing characteristics of Si_3N_4(HP) shown in Figures 8 and 9, the processing speed does not increase with the increasing diameter of the particles. The reason for this phenomenon may be due to the lowering of the grinding particle carriage capacity of the processing liquid (water) and a decrease in the number of grinding particles that act on the processing surfaces as the size of the grinding particles increases. Namely, if the grinding particles are uniformly placed on the processing surfaces in one layer, the increase in the particle's diameter results in a decrease in the number of grinding particles per unit area.

Therefore, in this respect, it is believed that a narrower particle size distribution is advantageous.

When the materials for semiconductors and electronic parts are processed, the use of SiC or Al_2O_3, characterized as having low strength, is advantageous in most cases although, there is a drawback in terms of the processing speed. For these materials, depending upon the characteristics of the grinding particles and the selection of the grinding particle size, the electrical characteristics following processing may be influenced.

Several of the author's examples will be discussed with regard to the contents mentioned above. In order to investigate the impact crushing processing method in more detail, statistical phenomena should be looked at

through sufficient data. In fact, since the grinding particles have uniform particle size distribution, test samples were prepared as much as possible, and a summary of the results was obtained.

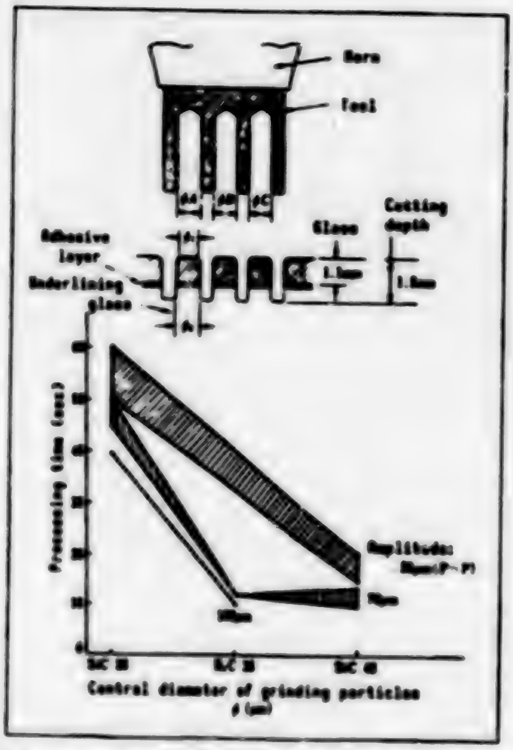


Figure 10. Grinding Particles (Diameter) and Processing Speed

In Figure 10, the tool was made from a mild steel square-shaped bar by placing three holes, ϕ1.3 mm (A,BE,C), parallel to the axial direction. Three cylindrical test samples, ϕ1.3 mm, were cut out from a 1.1 mm thick glass plate simultaneously. Processing was carried out 20 times, changing the processing conditions. Although a complete comparison cannot be made since different kinds of grinding particles were employed, the general trend can be seen.

Within the limits of the central diameter of the grinding particles of ϕ28 - 48 μm and the tool's amplitude of 50 - 100 μm, the processing speed varied from 1.5 to 9 mm/min. From the aspect of the processing speed varied from 1.5 to 9 mm/min. From the aspect of the processing speed, the larger grinding particle diameter and tool amplitude bring better results. In addition, the use of grinding particles with high fracture strength, such as B_4C, stabilizes the processing speed fluctuation and, therefore, is advantageous.

The relationship between the diameter of the grinding particles and processing clearance is shown in Figure 11. The processing clearance was obtained from the following procedures. The diameter of all 20 test samples prepared under the specific processing conditions were measured by

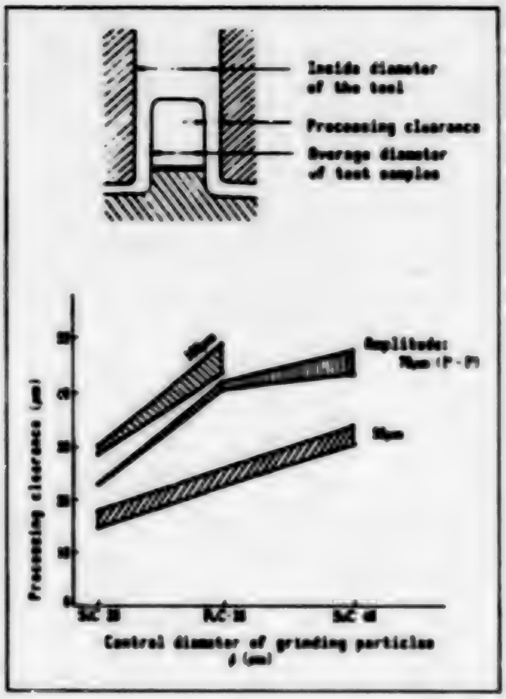


Figure 11. Grinding Particles (Diameter) and Processing Clearance

correlating them to the holes of tools A, BE, and C. Their average diameter was obtained, and then the processing clearance of A, BE, and C was calculated from the equation.

(Inner diameter of tool - Average diameter) x 1/2

The processing clearance becomes larger as the grinding particle diameter and tool amplitude increase. It is thought that the increase in the tool amplitude tool activates the circulation of the grinding particles, and the grinding particles with larger diameters, migrating around the tool, enter the processing surfaces more actively. However, the processing clearance fluctuation becomes the smallest when the tqol amplitude is 70 μm.

Table 2. Tool Amplitude and Dimensional Differences

Amplitude of tool		50 μm	70 μm	100 μm
		A B C	A B C	A B C
Grinding particles	SiC ϕ28 μm	5 15 10 Average 10	15 15 10 Average 13	10 15 10 Average 11.6
	SiC ϕ48 μm	5 15 15 Average 11.7	15 10 20 Average 15	
	B_4C ϕ38 μm		5 10 25 Average 13	20 20 20 Average 20

(Unit: μm)

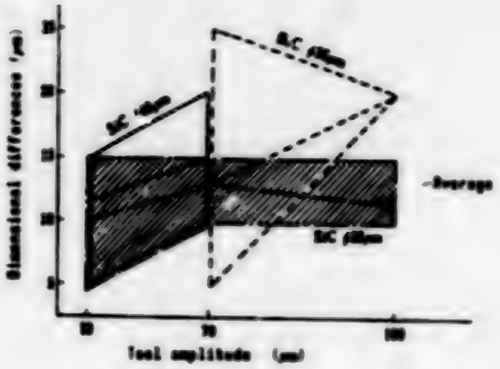


Figure 12. Tool Amplitude and Dimensional Differences

Table 2 and Figure 12 show the relationship between the tool amplitude and dimensional differences obtained from the above test samples. The dimensional differences shown here lie between the maximum and minimum diameters obtained from measuring the 20 cylindrical test samples that correspond to the holes A, BE, and C of the tool.

For grinding particles of SiC $\phi 28$ μm, the dimensional difference is 5 μm, and the average is 10 μm when the amplitude of the tool is 50 μm. Relatively good results are obtained. However, when the fluctuation ratio of the dimensional difference is observed.

$$\frac{\phi_{max} - \phi_{min}}{\phi_{min}} = 100 \text{ (percent)},$$

an amplitude of 70 - 100 μm produces the small figures in this ratio.

Similarly, when the tool's amplitude is 70 - 100 μm, a small fluctuation ratio of the processing clearance results.

Furthermore, in the relationship between the processing speed and the fluctuation ratio, an amplitude of 70 - 100 μm does not require a long processing time, and stable processing can be accomplished.

Therefore, in pursuing processing operations with good reproducibility, a tool's amplitude of 50 μm is too small for grinding particles $\phi 28$ μm in diameter. As a result, the circulation function of the grinding particles is degraded. It is believed that its processing conditions are very unstable.

From the viewpoint of maintaining precision alone, the use of small grinding particles without changing the tool's amplitude may be more effective for this case. The alternative is that the maximum permissible limit of precision be enlarged, and processing be carried out at an amplitude of about 70 μm. This shortens the processing time and improves the stability of the operation.

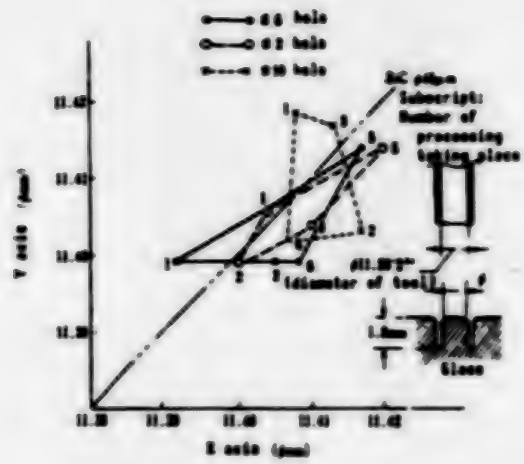


Figure 13. Results of Measuring Hole Diameters

Figure 13 shows the measured results of circular hole processing. Ten stainless steel pipes with inside diameters of 11.58 mm are placed on the horn with an edge surface diameter of 90 mm. Measurements were conducted following several processing passes. Hole numbers 6, 2, and 10 are used to avoid confusion involving the locational relationship between the tool and the processed holes. The measurement value is the dimension of the hole entrance. The processing device employed has 16 kHz and an output of 1 kW. The grinding particles used are of SiC of ϕ40 µm. The processing speed is about 0.5 mm/min.

Several factors influence the processing precision. They are: 1) tool abrasion (worn uniformly in both the longitudinal and radial directions); 2) the vibrational configuration of the tool (the existence of the tool); and 3) the effectiveness of the grinding particles (grinding particles uniformly supplied to all tools in all circumferential directions). The tolerance is within the limits of ±0.02 mm at the processing entrance.

Figure 14 shows the measured results of cutting a ϕ3 mm cylinder from a glass plate. The tool is made from a S45C pipe with an inside diameter of 3.12 mm. It is finished by lapping. The device used has 16 kHz and an output of 150 W. The cutting depth is 5.5 - 6.5 mm. The processing speed is 1.3 - 4 mm/min. This figure indicates a typical example representing 40 test specimens.

Grinding particles with diameters of 28 µm naturally exhibit less slackness and inclination at the processing entrance when compared to those with diameters of 48 µm. Their inclination is about 25 µm/5,000 µm = 5/1,000 for grinding particles with diameters of 28 µm, and about 40 µm/5,000 µm = 8/1,000 for those of 48 µm.

5. Problems To Be Solved in the Future

The development of abrasion resistant materials for the tool is one subject to be solved. At this moment, the sintering of synthetic diamond is promising for limited application.

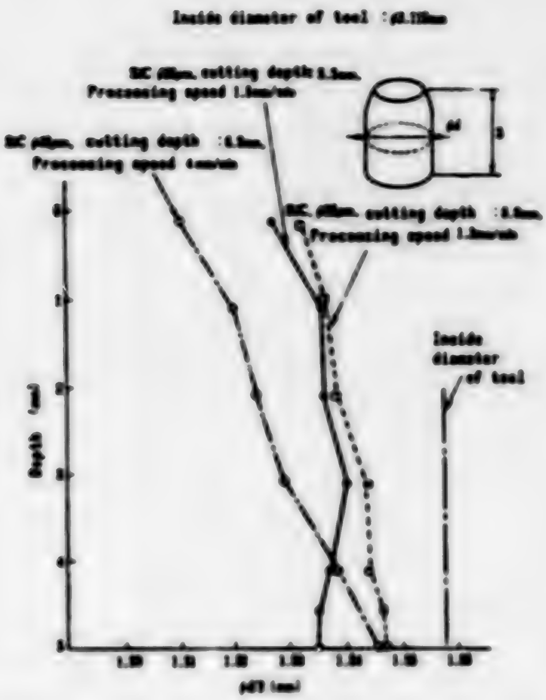


Figure 14. Cutting of Glass Cylinder

In the case of the processing of ferrite, the cutting ratio (cutting depth amount of abrasion in the longitudinal direction) exceeded 10^4 for spot facing with a depth of 2 mm and diameter of 10 mm. The processing precision range from 10.80 to 10.83 mm in diameter at 0.5 mm above the bottom of a processed hole. More than 80 percent of the diameters of all processed holes were within 10.81 - 10.82 mm. Regarding the precision of a hole depth, more than 70 percent of all processed holes ranged from 2.00 to 2.03 mm. The device used for this processing had an output of 300 W, with a depth control function. Its processing depth was set at 2.0 mm, and the grinding particles were SiC of ϕ48 μm. The slickness of the tool at its corner and abrasion at other locations were hardly observed.

In addition, a processing ratio of more than 10^4 was accomplished for a hole processing example with a diameter of about 1 mm. For the case of ceramics, which are relatively easily processed, a processing ratio of more than 10^3 has been obtained for hole processing with a diameter of about 1 mm. This material is certainly an excellent material for processing. If limitations on processing and heat deterioration are overcome by an applied device, this material can be utilized for varying purposes.

6. Conclusion

Even in the field in which the ultrasonic processing method has been applied effectively and positive achievements have been seen, there is still room for improvement. The shapes of the horn and tool are factors which figure prominently in this method. Certain processing procedures will correspond well to this method.

In most cases, however, people believe that the tool and horn must be prepared for a particular processing purpose, and they demand a resonant system that bears intensive resonance with good efficiency. For this reason, at first glance this method appears to pose problems. To be sure, certain problems must be solved in order to prepare a stable horn and tool. In fact, it is necessary to pay attention to several aspects. The problem associated with residual cracks on the processing surfaces that relates to an improvement in processing quality is one of these difficult problems requiring resolution.

With regard to these problems, it is believed that they can be grasped by observing many processing examples, and their data should be applied at this stage.

The author, who has experience not only in manufacturing the ultrasonic processing device but also in ultrasonic processing itself, hopes that this paper will aid readers in some respect.

Pressure Molding Technology

43064051 Tokyo KIKAI TO KOGU in Japanese May 88 pp 81-89

[Article by Takashi Kasai and Akihiko Hirota, Yuken Industrial Inc.: "Pressure Molding Technology for Fine Ceramics"]

[Text] 1. **Molding of Fine Ceramics and Pressure Molding**

Several molding methods exist for ceramics. They are:

(1) The method, well known for some time for molding pottery.

(2) The casting molding method, in which slurry raw materials are poured into a plaster mold and dried.

(3) The doctor blade method, in which slurry raw materials with constant thickness are poured continuously, and which is used for thin plate molding for making chip boards.

(4) The extruding molding method for molding cylinders or columns.

(5) The pressure molding method for molding powdered raw materials under pressurization.

(6) The injection molding method for melting ceramics into organic solvents and then pressurized.

In this article, item (5), the pressure molding method, will be discussed.

Regarding the various molding methods described above, "Research Study of the Development and Utilization of Machines Relating to Manufacturing of Fine Ceramics" conducted by the Japan Industrial Technology Promotion

Association in 1984 can be referred to especially for the survey of fine ceramics. According to this study, of 125 cases cited, about 60 percent involve the pressing method and about 20 percent the rubber pressing method (CIP: cold isostatic pressing), respectively. It can be said that these constitute the general molding methods.

The metal mold pressing method and rubber pressing method, classified as pressure molding methods, can be used not only for fine ceramics, but also for powdered metal and ferrite, although minor differences exist in their characteristics.

Fine ceramic products (parts) of good quality have the following attributes in addition to their individual specific characteristics.

(1) Density is very close to that found theoretically.

(2) Almost no internal and external defects exist.

Therefore, the following are demanded for molding processes in addition to cost:

(1) Easy baking (for degreasing purpose)

(2) Less grinding processing (grinding margin and time) required after baking.

Item (2) is the main subject of this article. Research and development is being vigorously carried out from the aspect of precision and efficiency. It is needless to say that the raw material characteristics comprise an important factor.

The following are demanded for pressure molding:

(1) That the molding density be uniform, with only a few nonhomogeneous strains during baking.

(2) That no cracks occur during molding.

(3) That little scattering occur in shapes.

(4) After molding takes place, when considering a primary processing before baking, the strength (green strength) to bear a chuck force during lathe processing must be assured.

(5) Impurities (contamination) not be easily mixed.

(6) Arrangements for mold modification and alternate operational conditions be easily carried out.

The molding pressure of the pressure molding method introduced here is realized by the metal mold pressing method or the rubber pressing method. In either case, a molding pressure of 1,000 - 2,000 kgf/cm^2 is generally used

for fine ceramics (for the case of powdered metal molding, a pressure of 4,000 - 7,000 kgf/cm^2 is commonly used).

2. Metal Mold Pressing Method

The pressing method in which powder is molded by using a metal mold composed of an upper punch, a lower punch and a motor is called the metal mold pressing method.

2.1 Outline of the Device

Metal mold pressing can easily and continuously automate a series of processes, from the filling up of powder to extracting molded products. When compared to the rubber pressing method, it is more conducive to mass production. It is possible to obtain a smoothly finished surface that does not require additional post-processing by preparing the surface of the metal mold according to this method. In addition, once the stroke of a punch is set up, the reproducible dimensional accuracy of a molded product can be realized and, in addition, once the pressure is determined, a molded product with uniform density can be made.

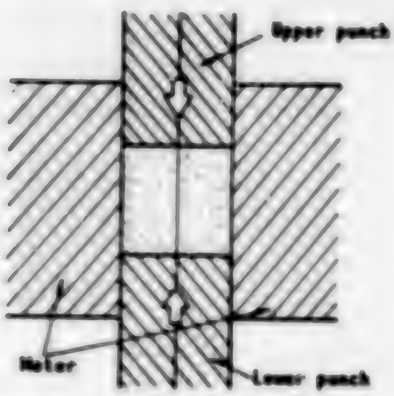


Figure 1.1 One-Axial Pressurization

However, since it is the one directional (axial) pressurization method (Figure 1.1), friction between the powder and the metal mold occurs, and nonuniformity in the density distribution of a molded product is easy to see. Therefore, limits have been placed on the shape and dimensions of the molded product. For example, in the case of a cylindrical molded product, its height should be less than twice its diameter.

There are two types of metal mold pressing--one is oil pressured pressing and the other is mechanical pressing. They are further classified into single-acting pressing, double-acting pressing, and multistage pressing, depending upon the operational method of the metal mold (Figure 1.2).

Single-acting pressing is the pressing molding method employing a fixed lower punch during molding. When a motor is fixed, it is termed the single-pushing-type pressing method. When the motor is supported by a spring and is pushed down by a frictional force generated between powder and the motor

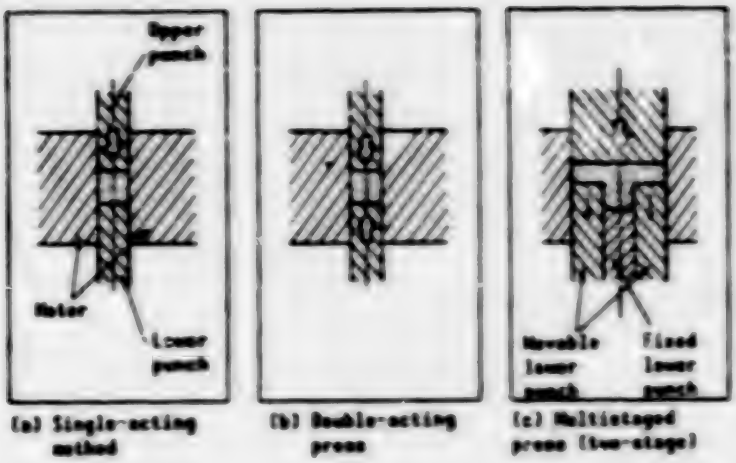


Figure 1.2 Classification of Metal Mold by Operational Method

during molding, it is called the die floating method. On the other hand, when the motor is forced down with a controlled speed, it is called the withdrawal type. When the motor descends during molding, friction between powder and the metal mold can be reduced. The occurrence of a low density portion at the lower punch side, which is seen during single-pushing-type molding, can be improved, and a product of better quality can be obtained.

Double-acting pressing is a type of pressing molding method in which powder is molded by operating both the upper and lower punches with a fixed motor. By pressurizing powder from both the upper and lower sides, the occurrence of a low density portion in a product can be improved. For instance, when the upper and lower punches are controlled to make the same stroke at the same speed, the low density portion of a product migrates toward the center portion. As a result, a product with symmetrical density distribution with respect to the upper and lower planes can be made.

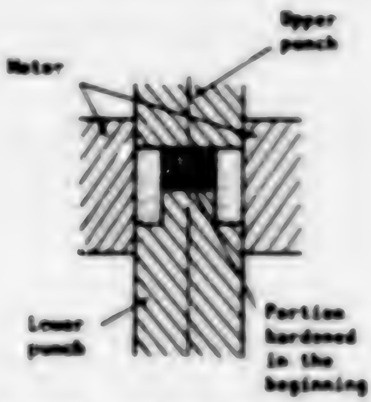


Figure 1.3 Uniform Density Caused During Molding of Product With Staged Shape

The multistage press has a movable lower punch supported by a spring on the double-acting press previously mentioned. This press is effective for manufacturing a molded product with a several staged configuration. If this

movable punch is not used, a part molded with a small stroke is hardened in the beginning. The molding of a portion requiring a large amount of powder replenishment may be ended without if sufficient molding conditions are not present. This could cause nonuniform density (Figure 1.3). This tendency becomes more apparent when the molded product has a larger difference in a staged shape.

Next, when the mechanical pressing is classified by driving method, it is divided into the crank press, toggle press, and cam press.

Under the crank press, a rotary motion is altered to a rectilinear motion of a punch by the crank that is installed in an eccentric configuration.

The toggle press is also called the knuckle joint press. This combines the toggle mechanism with a crank.

The cam press is the most frequently used of the mechanical presses, since the punch motion can be freely altered by changing the shape of the cam in the press.

2.2 Molding Conditions of Oil Pressured Pressing

As mentioned above, there are several types of metal mold pressing methods. The configuration of automatic molding when using the withdrawal-type oil pressured press is shown in Figure 1.4. In these processes, a cylindrical product is molded by adding the core to the metal mold.

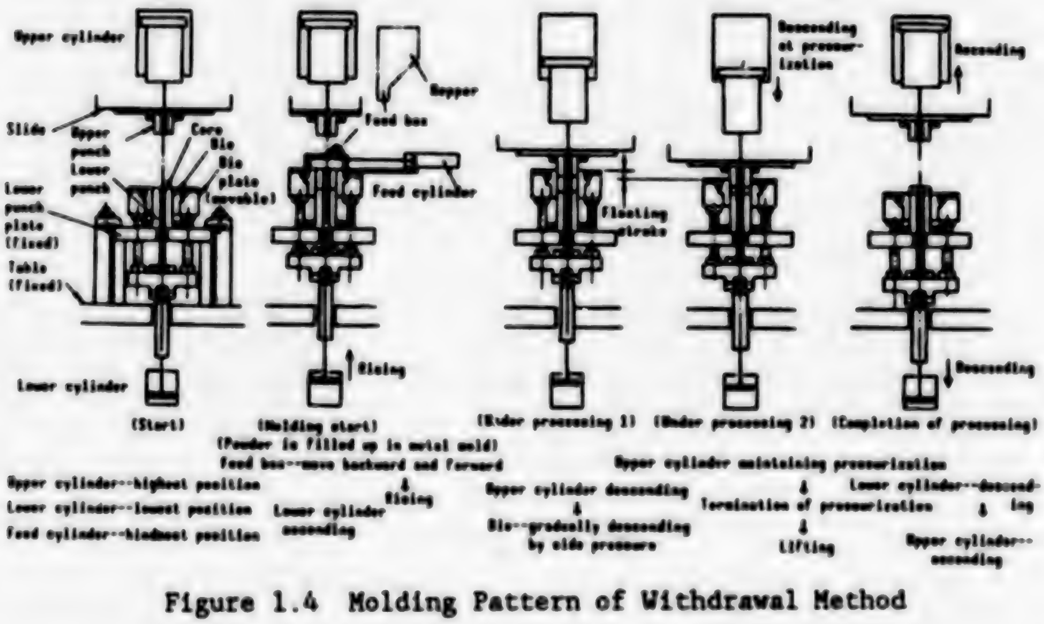


Figure 1.4 Molding Pattern of Withdrawal Method

The upper cylinder, the lower cylinder, and the feed cylinder are located at the highest point, the lowest point, and the hindmost point, respectively. When a signal indicating the start of molding is inputted, the motor rises to the replenishment point that had been set up from the start. At the same time, the feed box containing powder supplied from the

hopper carries out the shaking process, and powder fills up the cavity. When the cavity is deep, filling up of powder by raising the motor is effective in improving the filling up efficiency and relieving the bridge phenomenon of powder.

After the powder-fill operation is completed, the feed box descends to the hindmost position, and the level of the motor is the same as that of the powder. If the upper punch is inserted then, powder flies up. Therefore, molding should take place after the motor is slightly above the top surface of the powder.

During molding, the motor is lowered due to the side pressure generated by friction and the impelled oil pressure. The upper punch descends until it reaches the position set up in the beginning or the pressure limits set. Then, molding takes place, and the pressure is maintained for several seconds.

After that, the product is released from the mold when the pressure of the upper cylinder is released, and the upper punch is lifted according to the resilience of the molded product. Usually, the motor descends before the upper punch ascends.

This is one molding cycle. For totally automatic operations, such pre- and post-devices as the powder supply device to supply powder to the hopper and the molded product detachment device are attached.

Next, problems that occur during actual molding processes and their countermeasures will be discussed. The problems discussed here are associated with the crack and fracture of molded products.

These problems seem to be influenced mainly by the characteristics of the powder itself, such as the moldability and flowability, instead of the press features. Actually, a powder exists that cannot be molded despite the kind of special process applied. On the other hand, some powder can be molded in different ways depending upon the molding method used.

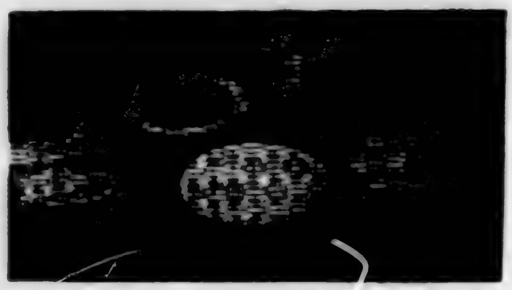
Regarding cracks and fractures, two different kinds exist. One is a longitudinal crack in the upward and downward directions with respect to the molded product, while the other is a lamination crack that appears in the lateral direction.

The longitudinal crack occurs when a molded product is smashed by the weight of the upper punch during its extraction. In order to suppress this phenomenon, the molded product should be released from the mold after the upper punch is raised as much as the amount of spring back of the molded product.

On the other hand, the lamination crack occurs when the upper punch is detached from the molded product after molding, and the molded product is released from the mold. Then, spring back in the lateral direction occurs at the portion released from the motor in a sequential manner. When the molded product is released from the mold by holding the molded product with

both the upper and lower punches with moderate force, the lamination crack can be eliminated to a certain extent.

It is important to provide the detailed control mechanism described above for the metal mold press. Oil pressure pressing is characterized by its facility for controlling this mechanism.



Photograph 1.1 Example of Molded Product by Metal Molding Press

Lastly, an alumina molded product processed by the withdrawal-type oil pressure press is shown in Photograph 1.1. The surface of the product is very smooth and fine. This is a characteristic of the metal molding press. The longitudinal stripes observed on the side wall of the product are a result of the adhesion of abraded powder microscopically scraped off from the inside of the motor at the release of the product from the mold, since the ceramic particles are usually harder than those of the metallic materials used for the metal mold.

3. Rubber Pressing Method (CIP)

3.1 Outline

CIP (cold isostatic pressing) is used to fill a mold made of elastic material, such as rubber, with powdered materials, and to soak it in the solution. Then, pressure molding takes place starting from the circumference. Since rubber is used for the mold, this method is also called the rubber pressing method.

This method has been widely used among industries since the latter half of the 1960s. It was first used for molding fireproof materials and superhard alloys. After that, due to the adoption of the continuous casting method in iron and steel manufacturing in the 1970s, this method was utilized for molding fireproof materials (ϕ100 x 1,500ℓ), such as soaking nozzles. The device has become large, and the number of devices has increased rapidly.

A typical example of a device produced in commercial quantities through this method is an insulator for automobile sparkplugs. The total number of these parts produced per year can be estimated by multiplying the total number of cars manufactured per year (15 million/year) by the number of cylinders per engine (4- or 6-cylinder).

3.2 Outline of Device

As shown in Figure 1.5, the rubber mold containing powder is placed in the high pressure vessel, and is then soaked and pressurized. The device for this processing method is composed of a high pressure vessel that can stand a high molding pressure, a high pressure generation device, the vessel's cover opening and shutting device, an oil pressure device to drive these devices, and a control device.

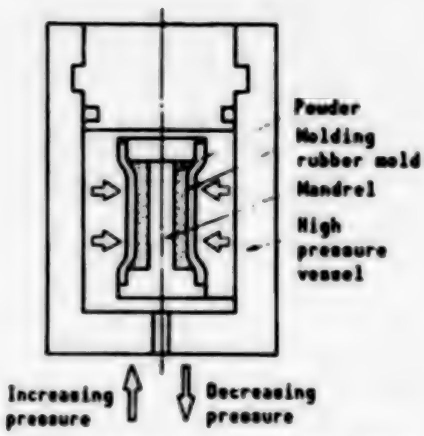


Figure 1.5 Principle of CIP Molding (Wet-Type)

This method can be used in two different ways. One is the wet method in which the rubber mold is soaked directly in the solution and pressurized, as seen in Figure 1.5. The other is the dry method in which the rubber mold is pressurized through the pressure rubber. The former is appropriate for molding products in different shapes. The latter is fitted for mass production, since molding takes place without the rubber mold contracting the solution directly. Therefore, the filling process, the insertion of the rubber mold, and the release of the product can be completely automated.

In both the wet and dry types, the high pressure vessel is designed to withstand fatigue caused by repeated stresses. An ingenious device is applied to the high pressure seal of the cover portion.

Regarding the high pressure generation device, the pressure increasing machine is the core part of the system. The primary pressure is oil pressure, and the secondary pressure involves water (wet-type) or oil (dry-type). Depending upon the size of the vessel, either the reciprocating type or the single pushing type is used.

The fluid injected into the high pressure vessel by the pressure increasing machine compresses powder through the rubber mold. It eventually reaches the necessary pressure due to the compression of the fluid.

The compressibility of the fluid is,

$$\beta = -\frac{1}{V} \cdot \frac{dV}{dp}$$

and its reciprocal number, i.e., the bulk modulus, is expressed by,

$$K = \frac{1}{\beta} = V \cdot \frac{dp}{dV}$$

Since the compressibility of water is $\beta \fallingdotseq (4\text{-}5) \times 10^{-5}$, and that of oil is $\beta \fallingdotseq 6 \times 10^{-5}$, when the pressure is increased to 2,000 kgf/cm^2, the volume change ratio becomes,

$$\frac{dV}{V} = \beta \cdot dp$$

and, substituting $dp = 2 \times 10^3$ kgf/cm^2,

$$\frac{dV}{V} \fallingdotseq (4\text{-}6) \times 10^{-5} \times 2 \times 10^{3} = 0.04 - 0.06.$$

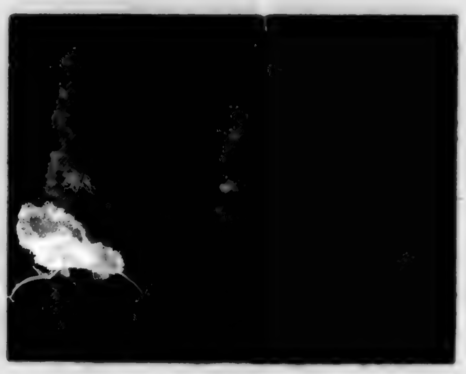
There is a decrease in volume by 4-6 percent. The volume added due to this decreased volume and the contracted volume of powder becomes the necessary amount of fluid to be supplied for molding. The size of the pressure increasing machine can be determined from this volume.

3.3 Conditions of Molding and Processes

In designing ceramic parts, the dimensions of the final configuration are set, and the dimensions for baking, including a grinding margin, are determined.

Contraction by the normal pressure baking of oxide ceramics is 15 percent for alumina and 18 percent for zirconia. These figures are relatively accurate. When the dimensional precision prior to baking is improved during mechanical processing, a reduction in the grinding margin and time can be reached.

Processing prior to baking (primary processing), such as cutting and grinding, is very easy when compared to processing following baking (secondary processing). For expensive raw materials such as zirconia, the cutting margin of the primary processing should be examined from the aspect of the loss of raw materials. From this viewpoint, designing of the rubber mold is carried out by taking into consideration the shape of the molded product and the molding compressibility of the powder.



Photograph 1.2 Molded Plugs (Left: Pressure Molding Product by CIP; Middle: Linear Grinding Product Prior to Baking; Right: After Baking)

As an example of a series of these processes, a comparison of the exteriors of automobile plugs at each stage is shown in Photograph 1.2. These are molded through dry CIP, primary processing (linear grinding) and baking.

The molding contraction of powder can be expressed as

$$1 - \frac{V_1}{V_0} = \beta$$

where, V_0 is the inner volume of a rubber mold and V_1 is the volume of a molded product. When this quantity is recognized as volume contractibility, it becomes 0.4 - 0.5 for alumina powder (spray dried) under a molding pressure of 1,000 - 2,000 kgf/cm^2.

Accordingly, when a sphere is molded, the contractibility of the diameter becomes,

$$D_1/D_0 = \sqrt[3]{0.6 - 0.5} \fallingdotseq 0.84 - 0.80$$

where, D_0 is the inside diameter of the rubber mold, and D_1 is the outside diameter of the product.

The dimensions of the rubber mold by volume capacity can be selected according to the required shape, such as a column and a cylinder.

In determining molding pressure, into only the characteristics after baking but also the conditions of the primary processing prior to baking should be studied.

When the processing of a molded body takes place, milling, holing, cutting, and grinding as the processing conditions, as well as the clamp and chuck methods of work, should be taken into consideration.

In particular, a workpiece must have enough strength to withstand its assigned job. This strength is influenced by the particle size and the amount of binders.

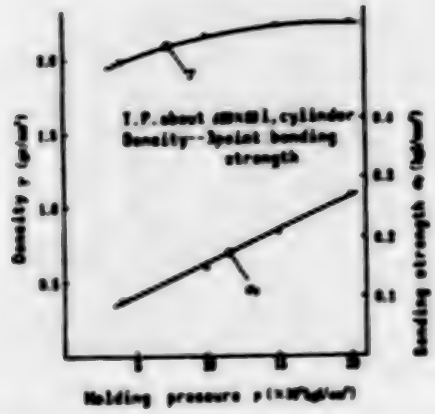


Figure 1.6 Molding Pressure vs Density and Bending Strength

There seems to be little data available for the systematic strength of a molded body. Nevertheless, Figure 1.6 shows the relationship between molding pressure and density and that with bending strength for a cylindrical test piece as an example. Note that the bending test was conducted using a ready device in order to see a tendency. It is not related to the bending testing procedures specified by JIS. These values may be referred to as reference data.

From this figure, the density becomes saturated with a certain molding pressure. When the secondary processing is required, it can be seen that as the density becomes higher, the chuck and transportation of a molded body are facilitated.

3.4 Molding Shape and Rubber Mold

In Photograph 1.2, at the bottom of the molded body on the left side of the photograph, the shape is stretched toward the outside, resembling the foot of an elephant.

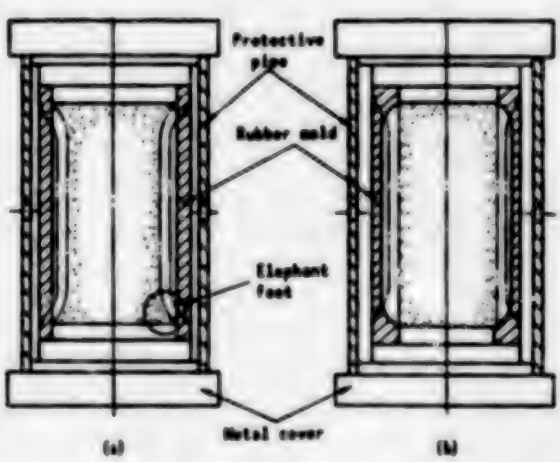


Figure 1.7 Examples of "Elephant Foot" Appearing in Straight Cylindrical Rubber Mold (a), and Rubber Mold (b) To Improve (a)

If a cylinder is used as an example, as shown in Figure 1.7(a), the shape of the molded body (straight shape) is deformed by pressure through the rubber mold as indicated by the dotted line, and at the bottom of the molded body, "the elephant foot" appears. If the shape of the rubber mold is modified, as indicated in Figure 1.7(b), this shape disappears and the straight shape of the molded body is obtained.

These shapes should be selected by taking into conside..ion the molding quantity, the allowable tolerance of the shape, and the life and cost of the rubber mold. When pressure is applied to the rubber mold, it is slightly deformed. Then, the powder is compressed and molded. During decompression, the deformation of the rubber mold is restored. However, when the molded body is released from the rubber mold, cracks may appear due to the friction between the rubber mold or the metal part (cover or mandrel) and the molded body, and the influence of the restoring speed vertically or radially. The particle size and the amount of binders may also exert an influence on the cracks. This phenomenon can be suppressed by lengthening the time for decompression to occur after the pressure has been reduced to a certain level.

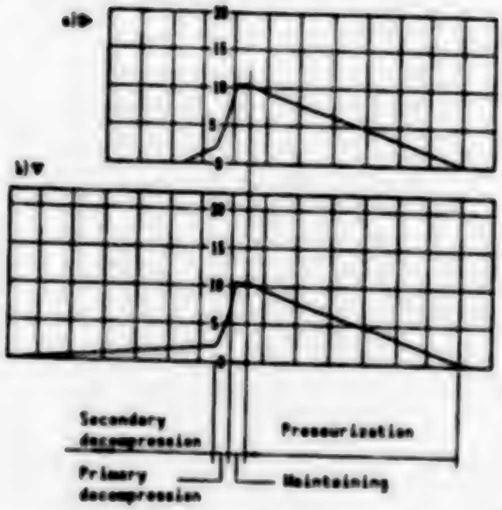


Figure 1.8 Examples of Pressurization and Decompression
(a) Secondary decompression 200 → 0 kgf/cm², 30 sec.
(b) Secondary decompression 200 → 0 kgf/cm², 3 min.

Figure 1.8 shows the actual record of pressurization and decompression. In this figure, the pressure was rapidly reduced to about 200 kgf/cm² (primary depression), then two different secondary depression times, 30 seconds, and 3 minutes, were used. Regarding the secondary depression time history curve, every company uses its own control method for changing or selecting the conditions. For the case of a large molded product, the secondary depression time is sometimes longer than 10 minutes.

Photograph 1.3 shows several examples of products molded by the wet- and dry-type methods.

Photograph 1.3 Samples Molded by Wet and Dry Rubber Presses

3.5 Wet-Type Rubber Pressing

Figure 1.9 shows the wet-type rubber press that is currently in general use for mass production. Its structure is such that the load exerted to the top and bottom covers of the high pressure vessel is sustained by a frame shaped like that of a window. A workpiece (molding rubber mold) is placed into the vessel by moving the frame.

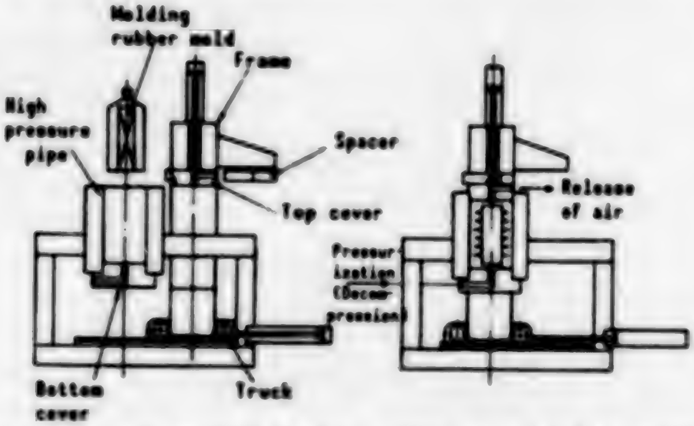


Figure 1.9 Wet Type Rubber Press With Mobile Frame

The wet type uses water (rustproof lubricant) for operation efficiency. The powdered raw material fills the rubber mold outside the device. The cover of the rubber mold is sealed by vinyl tape.

Depending upon the size of the vessel and the rubber mold, many products can be pressured and molded simultaneously. Since the shape of the molded product and its size are freely chosen in the wet-type rubber press, this method is usually applied to the molding of diverse products.

Photograph 1.4 illustrates the shape of the frame, and Photograph 1.5 shows an example of a small machine that manually rotates, opens and closes the frame for prototype experiments.

3.6 Dry-Type Rubber Press

Since a pressure rubber or diaphragm is placed outside the rubber mold, and pressurization takes place through it, the exterior of the molding rubber mold does not contact the liquid. This is why this method is called the dry

Photograph 1.4 Wet-Type Rubber Press With Mobile Frame (Pressure: 2,000 kgf/cm^2 Volume: ϕ1,000 x 3,550 mm)

Photograph 1.5 Experimental Rubber Press With Manually Rotating Frame

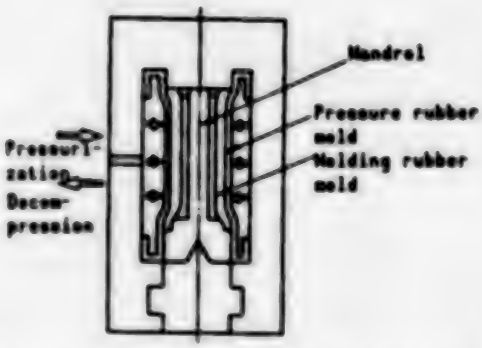


Figure 1.10 Principle of Dry-Type Rubber Press

type (Figure 1.10). Therefore, it is not necessary to change the pressure liquid from oil to water as is done with the wet type. When operated by oil pressure, the oil can be used as is.

Taking a rubber mold in and out of a high pressure vessel can be automated. Filling the rubber mold with raw material, and extracting the molded product can also be automated. Therefore, the complete automation of this method, suitable for mass production, is possible, similar to that of the metal mold press.

Figure 1.11 illustrates an automatic machine with four cavities per block. Different kinds of products can be molded by exchanging rubber molds corresponding to the desired shapes.

Photograph 1.6 shows the high pressure vessel of the dry-type rubber press with four cavities per block. Table 1.1 lists the characteristics of a metal mold press and a rubber press.

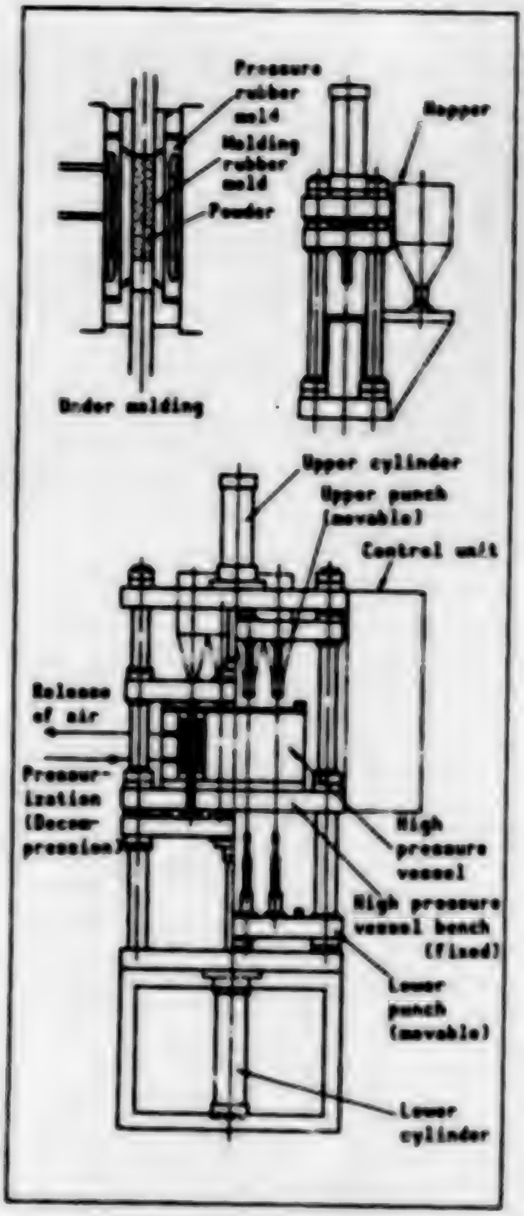
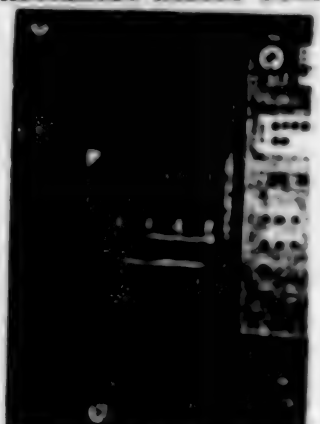


Figure 1.11 Dry-Type Automatic Rubber Press With Four Cavities



Photograph 1.6 High Pressure Vessel Dry-Type Four-Consecutive Shots Automatic Rubber Press

Table 1.1 Characteristics of Pressure Molding Method

Molding device		Shape of molded product	Dimensional precision of molded product	Cycle time	Mold cost
Metal mold press	Mechanical press	This product shapes include square, disk, segment shape, cylinder, column	$\frac{1}{100}$–$\frac{5}{100}$ mm	3~[illegible]sec	Expensive
	Liquid pressure press	Column, cylinder ($l/d < 2$)	$\frac{1}{100}$–$\frac{3}{1[illegible]}$ mm	[illegible]sec ~1min	Expensive
Rubber press	Wet type	Column, cylinder l/d : 1~[illegible] Crucible, bend	$\frac{1}{10}$–1mm	[illegible]sec ~[illegible]min	Several tenths that of metal mold
	Dry type	Column, cylinder l/d : 1~[illegible]	$\frac{1}{10}$–1mm	[illegible]sec ~[illegible]min	. .

4. HIP Molding

4.1 Outline

Hot isostatic pressing (HIP) was applied to industries about 10 years ago, with its application drastically increasing since 1981.

As its main use, this molding method has been effective for suppressing the internal defects of a molded product by processing sintered ferrite or superhard tools under a high temperature (1,000-2,000°C) and high pressure (1,000-2,000 kgf/cm^2) gaseous environment.

This method has also been applied to ceramics. For example, sintered parts are processed under high pressure and high temperature in order to eliminate voids, attempting to obtain a product with theoretical density. In addition, as a result of the work, new materials and products with different characteristics from those of conventional molded products processed by normal pressure sintering are being attempted.

Moreover, its application to the development of "directional materials," which combine the hardness and brittleness of ceramics and the elasticity of metal, has recently been observed.

Regarding gas environments, N_2 is added to a nitride system, and O_2 is added to an oxide system. These new processes aim at improving the mechanisms.

4.2 Outline of Device

The structure of the HIP is different from that of the CIP since pressurization is carried out inside the high pressure vessel by using high temperature gas. Therefore, precise plans and operational conditions are required in designing the high pressure vessel, high pressure gas generation device and heating furnace, as well as the method to detect pressure and temperature, etc., in accordance with the "high pressure gas handling standards."

This article presents only a simple introduction. Readers should refer to the device manufacturer's literature for details.

Injection Molding Technology

43064051 Tokyo KIKAI TO KOGU in Japanese May 88 pp 89-101

[Article by Toshiyuki Iwahashi, Niigata Steel Corp.: "Injection Molding Technology for Fine Ceramics"]

[Text] Injection molding technology for fine ceramics requires almost no post-processing for products, even though they have complicated shapes. Also, their precision can be assured. These are the characteristics of this technology.

The injection molding machine was originally used for the molding or forming of plastics. Since it has excellent productivity and manufacturing capabilities for uniformly molded products with good precision, these are combined with the powder sintering technique. However, since ceramic raw materials are pulverulent, fluidity and shapeability must be provided to make injection molding possible. For this reason, organic binders are usually added to the powder prior to the molding process. Therefore, the process to remove the binders becomes necessary. Since the removal of the binders is usually carried out by pyrolysis, it should be done by over a long period in order to prevent the occurrence of cracks due to the sudden change in temperature. This process is the weak point of the injection molding method. After the removal of the binders, sintering takes place in the usual manner. A sinter is then obtained.

Table 2.1 Manufacturing Methods of Fine Ceramics

Indirect method	Direct method
Pressure molding (pressing by mold, hydrostatic pressure)	Hot press method
	Melting casting method
Extrusion molding	Hot isostatic pressing
Injection molding	Synthetic simultaneous sintering (combustion sintering)
Liquid mud pouring	Casting (ceramics, ceramics-metal)
Turnery molding	Thin film method
Vibration molding (solid pouring)	Vacuum vaporization Sputtering Ion plating (PVD) Phase chemical reaction (CVD)
Doctor blade method	
	Reaction sintering method (Si_3N_4, SiC)

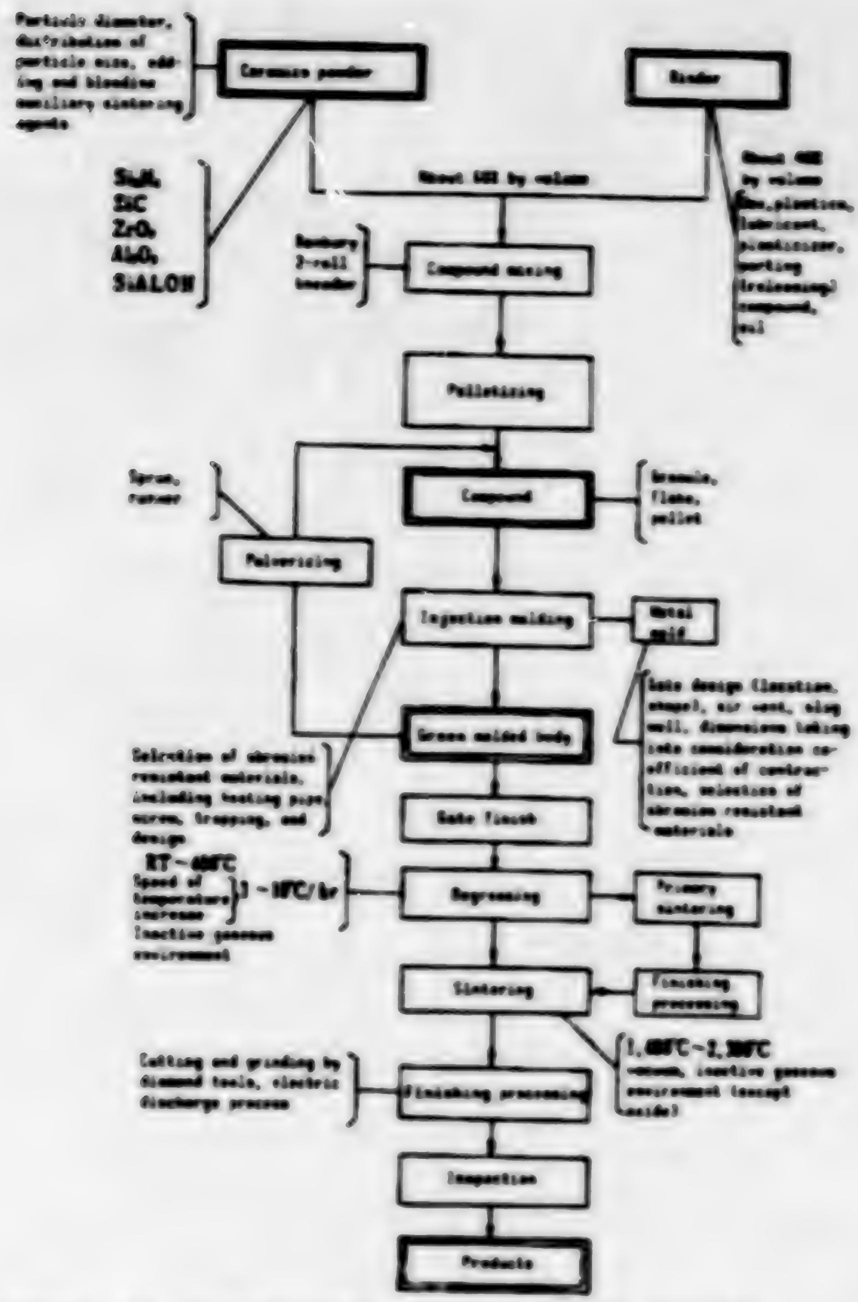


Figure 2.1 Manufacturing Process of Fine Ceramics by Injection Molding Method

Table 2.1 lists the fine ceramic manufacturing methods. Figure 2.1 outlines the manufacturing processes of fine ceramics by the injection molding method. It is believed that the adequate selection of products offering the advantages of the injection molding method and the improvement of factors causing increases in cost of metal modes and of the process to remove the binders will further expand the application of this method.

1. Characteristics of Injection Molding Method

The characteristics of the injection molding method applied to the molding of fine ceramic powder are listed in Table 2.2.

Table 2.2 Characteristics of Fine Ceramic Injection Molding Method

Item	Contents
Molding of complicated shapes	Molding can be done by one process for the shape obtained by a metal mold. It is particularly superior for three-dimensional structures. Finishing processing can be eliminated.
Dimensional accuracy Fine parts	The chance of sinter contraction after the removal of binders is large, but is less than 1 percent due to isotropy. For small parts, less than 0.5 percent accuracy an be expected.
High density High strength	Since high quality powder is used, and a molded product with uniform density and less strain is obtained, high density and high strength can be expected.
Productivity	The production speed of green molded bodies is better than that of other methods. Automation can be easily accomplished. However, plenty of facilities to remove binders are necessary.

This method is competitive with the press molding and liquid mud pouring listed in Table 2.1. However, the former is superior to the latter two in handling complicated shapes, dimensional accuracy, and manufacturing fine parts. Regarding the removal of the binders, the powdered raw materials, blending technique, and degreasing method have been improved. There has also been progress in reducing costs.

2. Ceramic Raw Materials

Table 2.3 shows typical fine ceramics used for injection molding. The diameter of the average particle of powdered raw materials is generally less than 1 μm, while that of sintered alumina is about 0.4 μm. The particles of raw materials obtained synthetically, such as zirconia, silicon nitride and silicon carbon, have smaller diameters. Therefore, the tendency exists to use a larger amount of binders since the comparative surface area becomes larger. Some raw materials (powder) require auxiliary sintering agents to be added. However, in some cases, such agents have already been added to the powder available to users. They include (calcia) for alumina, yttria for zirconia, and boron carbide (B_4C) for silicon carbide.

Table 2.3 Characteristics and Special Qualities of Ceramics

Materials	Alumina (Al_2O_3)	Silicon nitride (Si_3N_4)	Silicon carbide (SiC)	Zirconia (ZrO_2)
Characteristics	Excellent mechanical strength. High abrasion resistance, good stability for chemical environment	Highest strength and toughness among current ceramics. No deterioration up to 1,000°C. High impact resistance	No deterioration at high temperatures. Heat resistance up to 1,400°C. High hardness and high abrasion resistance. Used for heater materials.	High strength and heat resistance. Coefficient of thermal expansion similar to that of metal Used for composite materials. Applied to sensors.
Apparent specific gravity (g/cm^3)	3.94	3.15	3.15	5.7 - 5.8
Compression strength (kg/mm^2)	330	--	--	--
Bending strength (kg/mm^2)	55	65	45	60
Young modulus (kg/mm^2)	3.5×10^4	3.3×10^4	4.5×10^4	2.0×10^4
Hardness (45N)	88	87	91.8	85
Coefficient of thermal expansion, RT-800°C (/°C)	8.0×10^{-6}	2.9×10^{-6}	4.4×10^{-6}	11.0×10^{-6}
Thermal conductivity (cal/cm·s·°C)	0.065	0.037	0.158	0.005
Volume characteristic resistance (Ω·cm)	$>10^{14}$	$>10^{14}$	$10^3 - 10^6$	$>10^{10}$

5. Organic Binders

Table 2.4 lists examples of organic binders used for ceramic blending in injection molding. The binder types are classified as binding agents, plasticizers, lubricants, and other auxiliary agents.

Table 2.4 Binders for Injection Molding

Classification	Kinds of binders
Thermoplastic resin	PSE, PE, PP, SB, PMMA, PVAC, APPA, polybutyl acrylate, acryl nitrorubber, PEC, chlorine polyolefin rubber, polybudene, EVA, fluoric polyethylene, chlorosulfone polyethylene rubber, acetic cellulose, ethylene-ethylacrylate copolymer
Thermosetting resin	Phenol, formaldehyde resin, epoxy resin, polyurethane shellac, pitch metameric phenol resin, ureaformaldehyde
Water soluble polymer	PVA, polyvinyl acetal, fiber ether, EC, isobutylene-anhydride maleic acid copolymer, dextrine
Plasticizer	DEP, HPC, DBP, DOP (phthalic acid ester), fatty-acid derivatives
Lubricant	Stearic acid magnesium, stearic acid zinc, stearic acid aluminum, stearic acid diglycol, stearic acids or fatty-acid paraffin, wax
Other auxiliary agents	Natural animal and plant oil, salicylic acid, ethyleneglycol, silane vacuum oil, wood tar oil, pine root oil, asphalt, gas oil

3.1 Binding Agent

The purpose of binders is to preserve the shape of a ceramic powder molded body. Therefore, strength and elasticity are demanded. At the same time, a resin with low melting viscosity is required for good molding and releasing characteristics as well as for minimizing the required amount of additives. Accordingly, binders with low molecular weight are generally used. Their degreasing ability must be maintained. Olefin, styrene, and methacrylic acid resin are commonly used as binders. In order to prevent damage to a product during releasing and handling, these binders are sometimes replaced by an ethylene copolymer such as EVA. Wax is also used for this purpose. Microcrystalline wax is particularly superior in flexibility, strength, viscosity, and molding contraction. However, generally speaking, its strength to hold a molded body is inferior to that of resin. As the degree of crystallization increases in this material, the above tendency becomes more prominent. Nevertheless, its sliding, releasing, and degreasing abilities are good.

3.2 Plasticizer

Phthalic acid derivatives (DBP, DOP, DEP, etc.) are generally used. When these materials are added, the viscosity of a matrix decreases and the flexibility increases, as seen when they are added to plastics. At the same time, an improvement in the powder dispersion and degreasing ability is also observed. However, if a large amount of the plasticizer is added, it migrates to surfaces and degrades the releasing performance.

3.3 Lubricant

Paraffin wax (melting point: 57-63°C), microcrystalline wax (melting point: over 63°C) and polyethylene wax are available as lubricants. They also function as binders, and improve the lubrication of powder particles and binders. They are also effective in decreasing the load and improving the release from a metal mold.

3.4 Other Auxiliary Agents

The addition of coupling agents and titanate increase the binding force and affinity between powder and binders. The addition of sublimation materials improves the degreasing performance. An ingredient to alter the quality of a matrix is also added.

4. Blending and Mixing Method (Granulation)

4.1 Blending

In order to fill the gap between particles and to have an affinity for powder, a minimum volume of binders is required. Namely, if the apparent surface area of the binders is large, i.e., the diameter of the particle is small, a large amount of binders is needed. As shown in Figure 2.2(c), the necessary amount of binders for the moderate distribution of spherical particles is fewer than that for uniform distribution, shown in Figure 2.2(a). When the particle diameter becomes large and their distribution width becomes narrow, the state of dilatancy is shown in Figure 2.2(b), and degradation in fluidity occurs with the presence of shear stress during mixing and molding processes. Furthermore, when pressure is exerted, a dehydrating phenomenon occurs. This makes molding difficult.

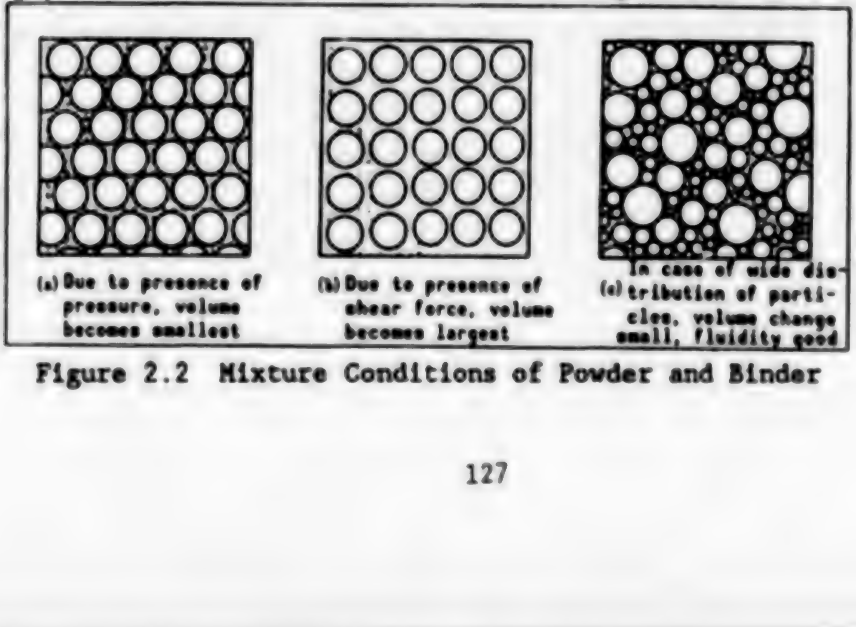
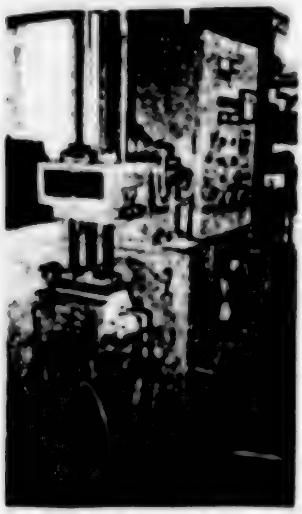


Figure 2.2 Mixture Conditions of Powder and Binder

An ideal blending ratio of powder and binders by volume is 60 to 40. For alumina with an average particle diameter of 1 - 0.4 μm, it is possible to conduct molding processing according to this blending ratio with 2-3 percent of the binder added by volume. However, for partially stable zirconia, it is necessary to increase the amount of the binder by about 10 percent (volume) when the average particle diameter of the binder is 0.3 μm and the apparent surface area is 18 m^2/g.

4.2 Granulation

A pressure kneader is generally used for mixing. The binder with a high melting point and larger particles is first thrown and melted. Then, powder is added. Finally, a plasticizer with a low boiling point is added. Mixing should take place for more than 30 minutes. Photograph 2.1 shows an experimental kneader with a capacity of 1 liter. Granulation is carried out by pulverizing the material following the cooling process for experimental purposes. Ideally, however, it is better to perform extrusion and pelletization to obtain pellets of uniform size. The direct mixing extrusion granulation machine is also available. Photograph 2.2 shows alumina pellets (ϕ2 - 3 mm).



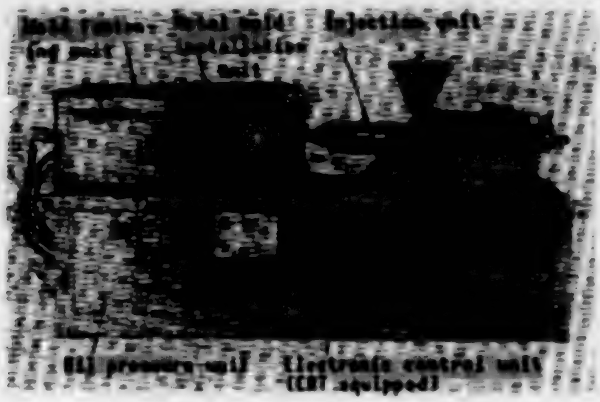
Photograph 2.1 Experimental Kneader With Capacity of 1 ℓ (made by Moriyama Seisakusho)

Photograph 2.2 Alumina Compound Pellet (Dai-Nippon Ink Chemical Green Seram AL9915)

5. Injection Molding Machine

5.1 Structure of Injection Molding Machine

The exterior of the injection molding machine is shown in Photograph 2.3. It is broadly divided into the injection unit, the mold fastening unit (metal mold equipped portion), the oil pressure unit and the electric control unit.



Photograph 2.3 Exterior of Injection Molding Machine (Ceramics Injection Molding Machine NNC50H: made by Nigata Tekkosho)

The injection unit stores raw materials. It is heated and controlled by the supply hopper and the external heater. It is composed of a heating pipe, made from abrasion resistant materials, and a screw. The rotation of the screw, and injection and feeding of the nozzle in the longitudinal direction are carried out by oil pressure.

The molding fastening unit fastens the mold during injection and opens and shuts the metal mold. It is operated by the oil cylinder.

The oil pressure unit is composed of a pump, which supplies high pressure oil to the mold fastening unit, the motor, the hydroblock, the control bulb to switch the flux, oil pressure and direction, and the instrument panels. If necessary, an accumulator using nitrogen gas is provided in the oil pressure circuit. The improvement of injection efficiency and the reduction of electricity are intended.

The electric control unit consists of the power supply, the motor control circuit, the heater control circuit and thermostat, the sequencer for operation control, the transfer, and the rectifier. Currently, the setting of conditions is done by the template key input method with CRT display, and a microcomputer is also incorporated. Various functions and mechanisms for the storage and resetting of molding conditions, the judgment of quality, and also monitoring and printing out of molding conditions are available. These correspond to FA. For providing a ceramic compound as a material exhibiting peculiar fluidic behavior, a delicate control mechanism is demanded depending upon the shape.

5.2 Molding Process of Injection Molding Machine

The general molding process of the injection molding machine is shown in Figure 2.3. Through the steps of mold fastening, nozzle forward movement and injection, the materials fill up the metal mold touch and the injection metal mold cavity. The contraction of the material is controlled and dimensional precision is preserved by maintaining pressure. Next, the screw is rotated, and the material is platicized. Then the necessary volume is

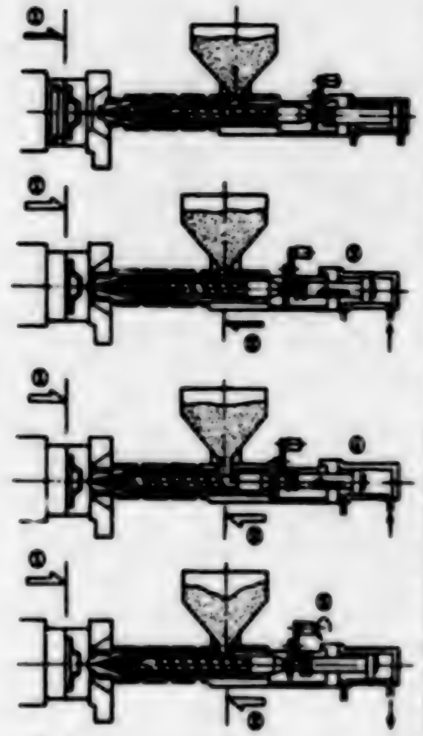


Figure 2.3 Molding Process

measured. This process covers the completion of the cooling of a molded product placed inside the mold. After the nozzle backs up, the mold is opened when cooling is completed. The molded product is thrown out by the ejector. This completes this process.

5.3 Complete Screw Material

The complete screw of the injection unit is an important part. It is designed to resist abrasion, which differs from requirements of the plastic molding machine. Material corresponding to glass fiber reinforced plastics has been available on the market. However, for ceramic molding materials, an improvement in abrasion resistance is desired, and efforts toward this end are being exerted.

(1) Material of Cylinder

The abrasion resistant cylinder currently in general use is the bimetallic cylinder. The inner side (a 2 mm thick layer) of the cylinder is constructed by diffusion joining of the abrasion resistant and corrosion resistant materials. Table 2.5 shows the components and hardness of bimetallic cylinders according to the manufacturer.

Table 2.5 Components and Hardness of Bimetallic Cylinders

Manufacturer (product)	Serial number	Hardness	Main components	Characteristics
WEXCO (United States) <WEXCO>	WEXCO 555	H_RC 50~55	Ni-Co-Cr-B	Abrasion, corrosion resistance
	WEXCO 666	H_RC 58~65	Fe-Ni-B	Abrasion resistance, low friction coefficient
	WEXCO 777	H_RC 52~64	Ni-Cr-B-WC	Abrasion, corrosion resistance
XALOY (United States) <XALOY>	XALOY 101	H_RC 58~64	Fe-Ni-B	Abrasion resistance
	XALOY 800	H_RC 50~64	WC-Ni	High abrasion resistance, corrosion resistance
PLASTEN Eng. (England) <BIMEX>	BIMEX AR-1000	H_RC 58~64	B-Fe	Abrasion resistance
	BIMEX HC-8000	H_RC 57~62	Cr-Fe	Abrasion resistance
	BIMEX PAC-9000	H_RC 40~55	Ta-C	Abrasion resistance, corrosion resistance
BRUX (England) <BRUX>	BRUX 100	H_RC 58~64	Fe-Cr-B	Abrasion resistance
	BRUX 600	H_RC 48~54	Ni-Co-B	Corrosion resistance
BERNA-BERNEX (Switzerland) <BERNEX>	A-110	H_RC 60~68	Fe-Ni-Cr-B	Abrasion resistance
	C-260	H_RC 49~53	Ni-Co-Cr-B	Corrosion resistance
REILOY METAL (West Germany) <REILOY>	REILOY 112	H_RC 64~67	Fe-Ni-B	Abrasion resistance
	REILOY 200	H_RC 58~62	Co-Ni-Cr-W-B	Abrasion, corrosion resistance
	REILOY 210	H_RC 61~65	Co-Ni-Cr-W-B	High abrasion resistance, corrosion resistance
Hitachi Metal (Japan) <H-ALOY>	H-500	H_RC 50~60	Ni-Co-Cr-Si-B	Abrasion, corrosion resistance
	H- 60	H_RC 58~65	Ni-Co-Cr-Si-B	High abrasion resistance, corrosion resistance
	H- 70	H_RC 58~66	Ni-Co-Cr-Si-B+ Special carbide	Super abrasion resistance, corrosion resistance
Nippon Seikosho (Japan) <NALLOY>	NALLOY 60	H_RC 58~64	Fe-Ni-B	Abrasion resistance
	NALLOY 80C	H_RC 53~65	Ni-Fe-Cr-B	High abrasion resistance, corrosion resistance
Kobe Steel (Japan)	FRC-C	H_RC 60~65	Co base alloy	High corrosion resistance, abrasion resistance
	FRC-2CV	H_RC 63~68	Co+ ceramics	
	FRW-N	H_RC 50~55	Ni base alloy	High abrasion resistance, corrosion resistance
	FRW-6NV	H_RC 63~68	Ni+ ceramics	

Even though a material exhibiting good abrasion resistance has been selected from among these materials, a certain degree of abrasion is inevitable. In particular, since mixing metal components with ceramic materials is not desirable, this situation should be avoided as much as possible. Instead, interior processing of the sleeve of the same ceramic materials is attempted by shrink fit. The amount of abrasion occurring inside the cylinder is shown in Figure 2.4. From this, it is seen that the use of ceramics in the feed zone is particularly effective. Lining zirconia along the overall length, sialon in the feed zone alone, and an abrasion resistant allow along the rest of the interior is being experimentally used.

Figure 2.5 shows the cross section of the cylinder. Under these circumstances, the screw and trap ring should be made from metal. Therefore, friction due to material rubbing is taken into consideration. The measured results of the apparent abrasion by the Daietu-type rapid abrasion tester are shown in Figures 2.6 and 2.7. From these data, it is judged that sialon surpasses others in terms of abrasion resistance. This

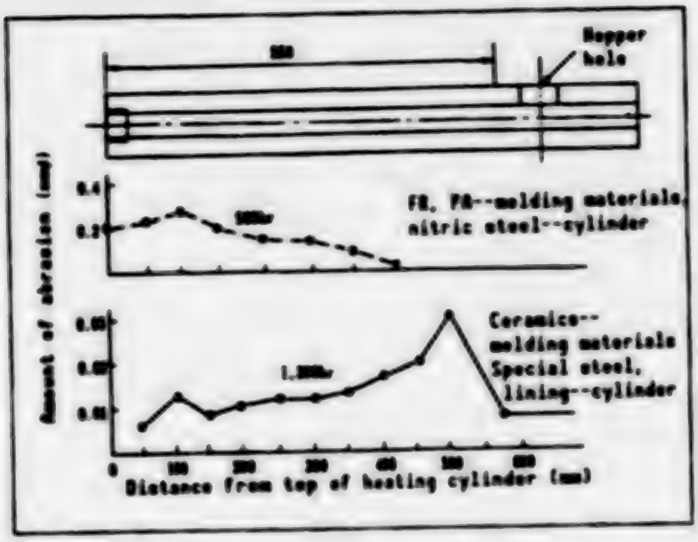


Figure 2.4 Cylinder Position and Amount of Abrasion

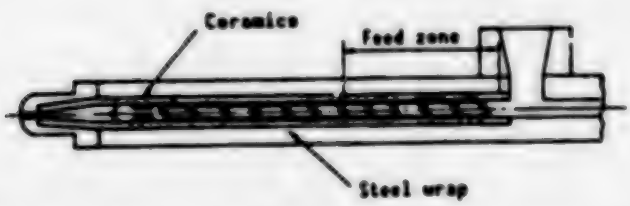


Figure 2.5 Cylinder With Shrink Fitted Ceramic Sleeve

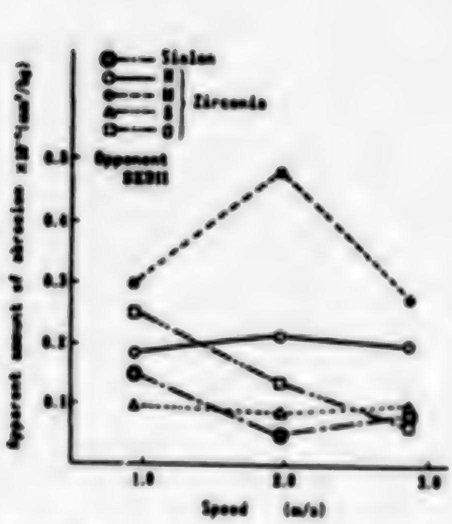


Figure 2.6 Sliding Abrasion Characteristics for SKD 11

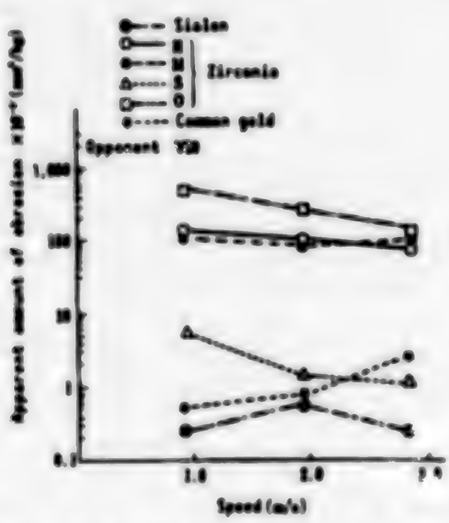


Figure 2.7 Sliding Abrasion Characteristics for KH-V50

material is also excellent in thermal impact resistance (ΔT sialon: 600°C, zirconia; 320-400°C, alumina; 210°C).

(2) Screw Materials

The places where abrasions occur on the screw are, as shown in Figure 2.8, 5-8 outer diameter threads within the feed zone, and 3-10 pitches of the inner diameter in the compression zone. In the tip of the metaling zone, not only abrasion but also corrosion is observed. The components of the abrasion resistant screw and hardness are listed in Table 2.6.

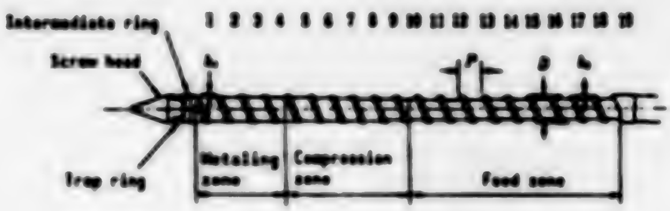


Figure 2.8 Screw Design

Table 2.6 Composition and Hardness of Abrasion Resistant Screw

Manufacturer	Serial number	Hardness	Main component	Characteristics
Mitsui Seiki	MSI	H_RC 58	Cr-Mo-C	Abrasion resistance, corrosion resistance
Hitachi Metal	YPT-4	H_RC 58~60	Cr-Mo-C	Abrasion resistance, corrosion resistance
	YPT-6		VC	Abrasion resistance
	YPT-7	H_RC 60~63	Cr-Mo-C-VC	Abrasion resistance, corrosion resistance
Nippon Seiko-sho	LS-2	(CM, 700) H_RC 60	Cr-Mo-C	Abrasion resistance, corrosion resistance
REILOY METAL	P-50	H_RC 58~62	Co-Cr-W	Abrasion resistance, corrosion resistance
ARENCO	Arnco-Alloy 20	H_RC 58~65	Co-C-Cr-W	Abrasion resistance, corrosion resistance

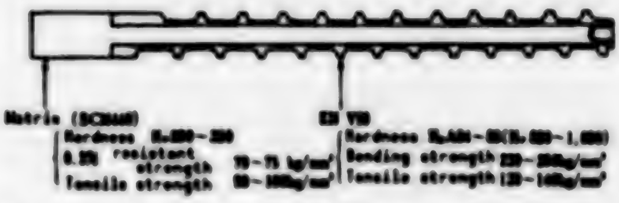


Figure 2.9 Structure of KH Screw and Mechanical Properties

As a measure to improve the hardness of the surface layer of the screw, a composite material of SCM 440 (steel) for the core bar and a V50 layer (made by Toyo Kohan) of the diffusion junction, made from SUS with detracted high hardness composite boride (Mo_2FeB_2) for the surface layer, is available (Figure 2.9). In addition, a several micron thick TiN and VC coating on the surfaces by the PVD and CVD methods is also effective.

Problems associated with its structural design include obtaining larger width of the screw top to reduce abrasion, and decreasing the P/D, L/D, and compression ratio (Cr) to reduce the torque. They have been adopted as needed.

(3) Ring and Nozzle To Prevent Counter Flow

The ring and nozzle are very important parts which affect the weighing accuracy of the injection molding machine. Specifically, the abrasion of and damage to the trap ring and intermediate ring would prove fatal to the machine. The careful selection of abrasion resistant and corrosion resistant materials and examination from the designing viewpoint are important (Figure 2.10).

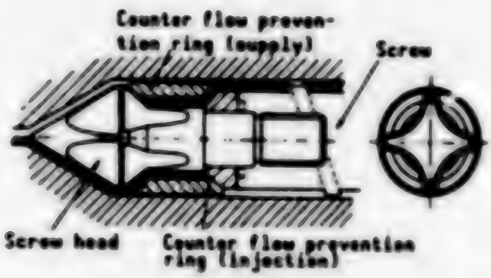


Figure 2.10 Ring Mechanism To Prevent Counter Flow

The causes of abrasion are the ceramic molding materials themselves and the grabbing and rubbing motions between the cylinder and the screw head. therefore, abrasion resistance and corrosion resistance are of particular importance. Figure 2.11 shows the abrasion resistant test results for the alumina compound of an Fe-base composite boride hard sintered alloy that is used for the above purpose. It illustrates the superiority of thin materials.

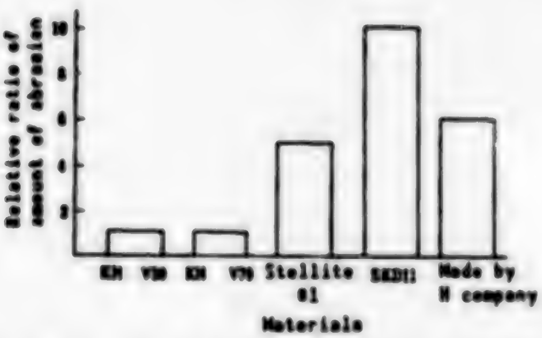


Figure 2.11 Abrasion Resistance Test Results

A similar measure for abrasion resistance is necessary for the nozzle. In addition, since heat generation by shear force occurs readily in ceramic materials when injection takes place, it is necessary to obtain a larger hole diameter than that for plastic materials.

6. Discussion of Design of Metal Molds

The melting fluidic behavior of ceramic molding materials shows that non-Newtonian fluid is similar to general polymeric materials.

The factors affecting fluidic behavior vary depending upon the particle size of the ceramic raw materials, the distribution of particle size, the melting viscosity of binders, and the amount of additives. Accordingly, it is necessary to study not only the molding conditions, i.e., temperature, velocity, and pressure, and metal mold design parameters, particularly gate position and shape, but also the characteristics of molding materials. Since the content of retained polymeric materials is generally larger than that of a single polymeric material, the compressibility is low, and the die's well is small when running into the cavity from the gate. In addition, jetting occurs easily. Since the cooling speed is generally fast, hardening is rapid, and the pressure falls faster than it does in simple plastics. This causes degradation during fusion and low strength.

Therefore, when compared to a plastic molding metal mold, the gate design becomes more important. The preparation of an air vent and slug well is also necessary.

6.1 Gate Design

A small gate is ideal, however, a pin gate usually causes jetting. This should be avoided as much as possible. The use of a fairly thick film gate and a ring gate enables a regular flow to be maintained, and causes little strain. The fan gate is preferable for a tab-gate system. However, when the spread angle of the fan becomes wider than 60°, the effect of the fan cannot necessarily be expected. This fact should be taken into consideration. The side gate is effective in preventing jetting since the flow strikes it once. However, a difference in the pressure conveyance between the surface of a wall close to the gate and that of an opposite wall is generated. Due to orientation and a difference in density, a molded product could be bent toward the gate side after baking. This should also be taken into consideration.

Gate Design Influence Exerted to Molded Products

Examples of molding are shown below. For the metal mold with four different kinds of gates on the strip-shaped cavities shown in Figure 2.12, the relationship between the gate design and the injection speed, temperature, and compounding prescription has been investigated.

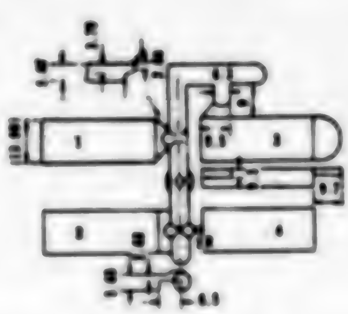
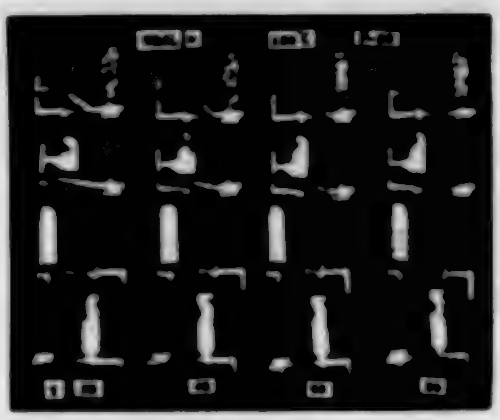
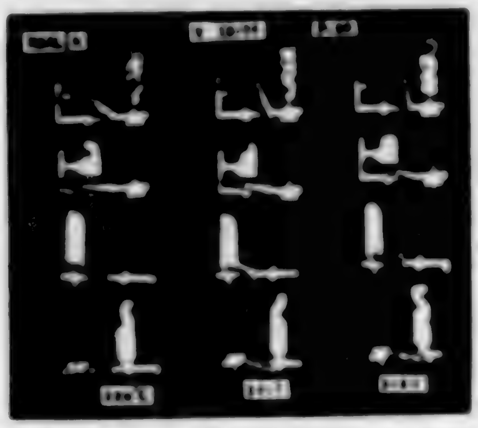


Figure 2.12 Metal Mold for Gate Design Study



Photograph 2.4 Influence of Injection Speed on Flowing State at Each Gate (alumina + resin binder) (Left: low speed; Right: high speed) (From top: pin gate, side gate, film gate, and fan gate)

The results of molding a resin alumina compound is shown in Photograph 2.4. Jetting could not be avoided regardless of how the temperature and speed were controlled at the pin gate. However, a good flow was seen when the thick film gate was used. Since the fan gate had a larger expanding angle (90°) than 60°, the opening section of the pivot performed the gate's duty. This caused jetting to occur. At the side gate, the flow hit the opposite wall, and stagnation appeared. As shown in Photograph 2.5, jetting was completely eliminated at 160°C.



Photograph 2.5 Influence of Temperature on Low Injection Speed (From left: 120°, 140°, 160°) (From top: pin gate, side gate, film gate, fan gate)

As the second example, the turbine blade type is shown in Figure 2.13. The influence on fluidic conditions exerted by the gate shape is shown in Photograph 2.6. Upon the evaluation of a baked body, the fan gate exhibited excellent results for molding materials with various viscosities.

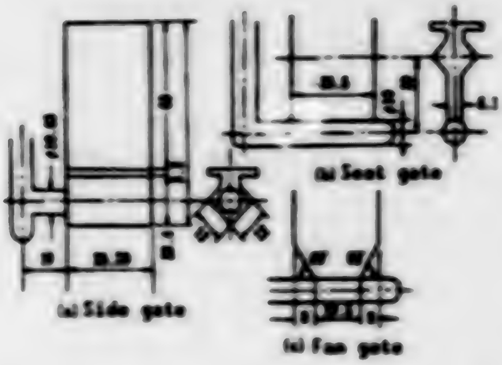


Figure 2.13 Gate Shape of Turbine Blade Metal Mold

Photograph 2.6 Flowing State and Three Kinds of Gates for Turbine Blade Mold (alumina molding material)

The third example is shown in Figure 2.14. A thick film gate and thick rectangular gate were adopted for the testpiece metal mold. As shown in Photographs 2.7 and 2.8, the prevention of jetting was successfully achieved by the use of alumina molding materials.

6.2 Liner Design

A short liner with a large cross section is arranged at sites equidistant from each cavity. A circular shape with a large ratio of cross sectional

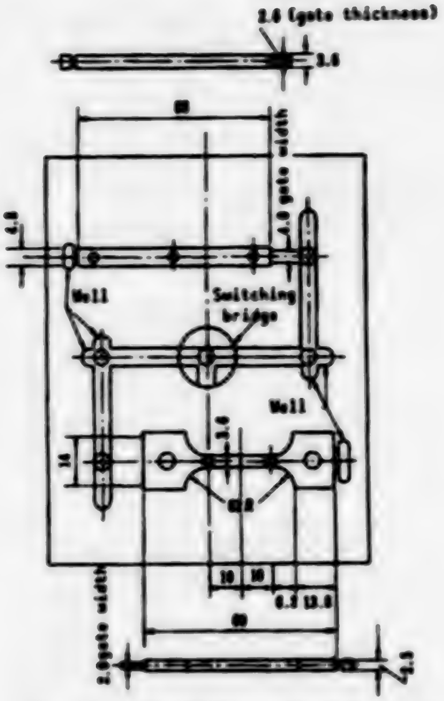
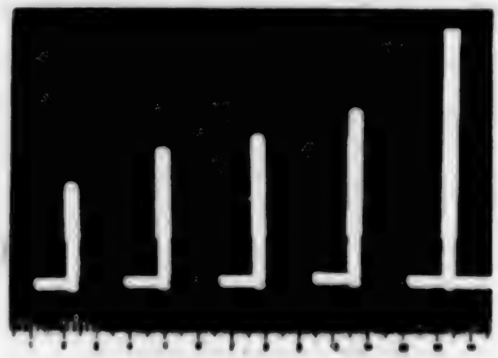
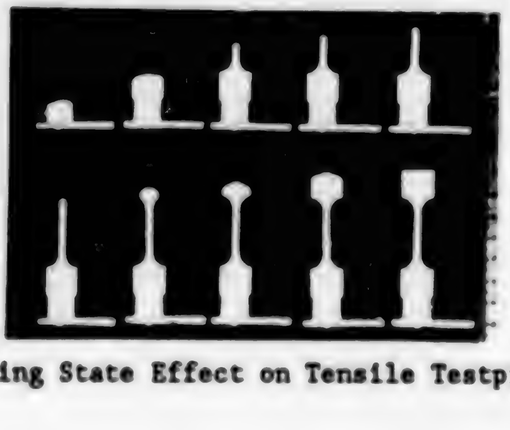


Figure 2.14 Drawing of Metal Mold Cavity for New Material Test Piece



Photograph 2.7 Flowing State Effect on Bending Testpiece Mold



Photograph 2.8 Flowing State Effect on Tensile Testpiece (for metal)

area to circumferential length produces good results with respect to processing capability, pressure loss, and thermal loss. However, due to processing capabilities, a trapezoidal shape is being widely adopted.

6.3 Cavity Design

There are two types of metal molds--one is the direct cutting type, and the other is the liner type. In selecting the dimensions of a final product, the total contraction ratio S(T) becomes the sum of the contraction ratio of a green molded body S(G), that of the binder's volatilization S(B) and that occurring during baking S(W),

$$S(T) = S(G) + S(B) + S(W) \quad (1)$$

Since the contraction shows isotropy, the following relationship exists between the volume contraction ratio S_V and the linear contraction ratio S_L,

$$S_L = 1 - \sqrt[3]{1 - S_V} \quad (2)$$

When the contraction ratio is small, the following approximation can be used,

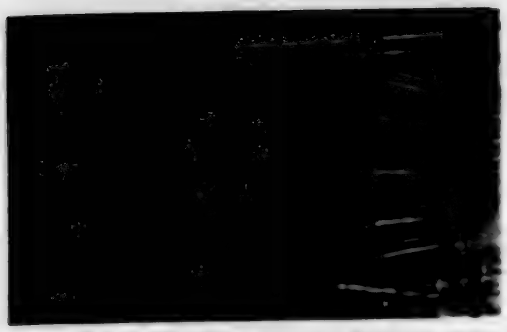
$$S_L = 1/3 \cdot S_V \quad (3)$$

S(B) in Equation 1 can be determined from the amount of binders added. Generally, it is about 40 percent by volume. This value is very large when compared to that of a green molded body S(G). This is because, since the dimensional precision of a sinter depends mostly on the quality of the green molded body, S(G) becomes important. In other words, the molding conditions to suppress surface shrinking due to contraction, internal voids, nonuniformity in density and orientation are selected, the linear contraction ratio S_L is experimentally obtained, and then the cavity dimensions are determined. This is the actual procedure, with S_L ranging from 10 to 20 percent. Its reproducibility is less than 1 percent.

6.4 Releasing Inclination, Etc.

Generally speaking, ceramic molding materials are inferior when releasing a molded product from a mold. The typical releasing inclination is more than 1/30 (2 degrees), twice as much as that of the mold for plastic molding. The angle of a spool is 5-10 degrees. This affects not only the cavity, but also the surface roughness. Therefore, polishing becomes important. As seen from the turbocharger rotor mold in Photograph 2.9, a separated mold structure employing a slide core is also effective in releasing a molded product from the mold.

In addition, the evacuation of air during the molding process should be fully considered. The preparation of an air vent of less than 0.02 mm that does not cause flash to occur and of a slug well that guides cold slug and the welding portion outside the cavity is also necessary.



Photograph 2.9 Turbocharger Rotor Metal Mold

6.5 Metal Mold Materials

The materials for the metal mold must have better abrasion resistance than those for the mold used for plastic molding. For both prototype and production type molds, a small quantity of varying kinds, with short life cycles and short development and production times, is demanded. Countermeasures involving the use of nonferrous materials showing better cutting capability but less durability, and the adoption of PVD or CVD coatings, such as TiN, to increase surface hardness are currently in use.

On the other hand, regarding the processing method, the technique to cut hardening materials by a direct cutting electric discharge processing machine is being applied. At the same time, the adoption of easy metal molds and unit metal molds is being promoted.

7. Molding Technology

In order to stably obtain a highly precise molded body with no defects, it is necessary to arrange the raw materials, metal mold, molding machine, molding conditions and auxiliary devices properly, similar to those for the molding of plastics. For ceramics, in particular, the selection of molding materials becomes a key to success.

There are several ways to evaluate flowability and moldability. These include viscosity measurements employing a flow tester, and measuring the plasticized state and flow length under practical conditions by installing a bar flow or spiral flow mold in the injection molding machine.

Figure 2.15 shows the relationship between shear speed vs. shear stress and viscosity for a resin binder blended with alumina molding materials. The flow tester was used for the measurements. Figure 2.16 shows the relationship between injection pressure and a spiral flow length as a function of temperature when the above material is processed by a spiral mold with a cross section of 10 mm (width) x 3 mm (thickness). Injection molding is possible if the shear speed inside the metal mold is 10^2 - 10^3 mm/sec, and viscosity is less than 10,000 poise. It is preferable that the viscosity be less than 5,000 poise, and the spiral flow length more than 20 cm.

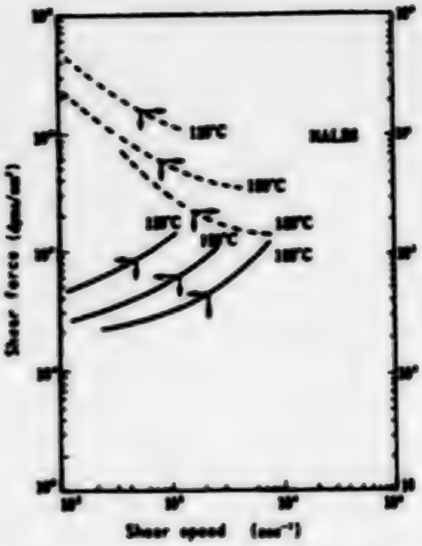


Figure 2.15 Flowing Characteristics of Acrylic Ceramics Compound

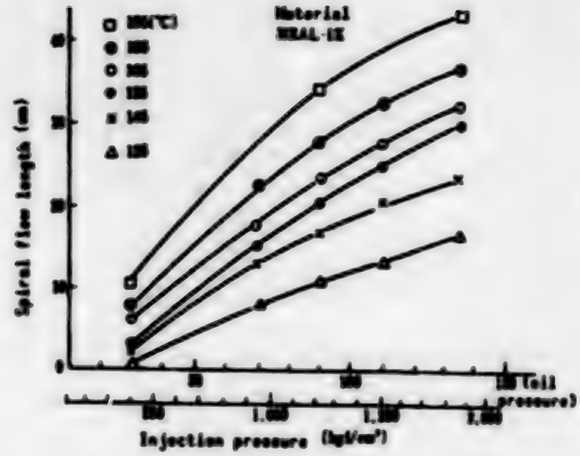
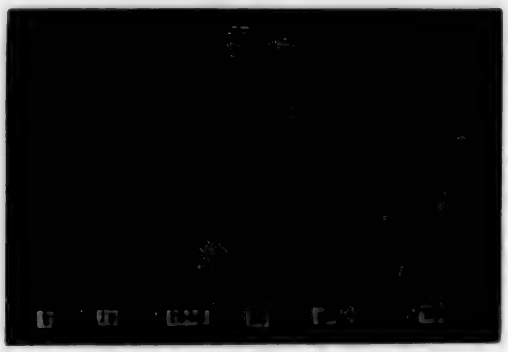


Figure 2.16 Relationship Between Injection Pressure and Spiral Flow Length



Photograph 2.10 Flowing State by Turbocharger Rotor Mold (Zirconia compound, Dai-Nippon Ink Chemical)

Table 2.7 Injection Molding Conditions of Turbocharger

Items		Conditions	Remarks
Ceramics		Zirconia	Weight: 85 g
Injection molding machine model		NDH100H3000	
Cylinder temperature	(Nozzle) N	155 (℃)	
	H_1	150 (℃)	
	H_2	145 (℃)	
	H_3	145 (℃)	
Metal mold temperature	Fixed	42 (℃)	
	Movable	42 (℃)	
Screw rotations		60 (rpm)	Screw diameter: 40 mm
Injection pressure		570 (kgf/cm^2)	
Pressure maintained		300 (kgf/cm^2)	
Injection speed		20 (mm/sec)	2-speed (8.5 sec)
Injection time		10 (sec)	
Cooling time		25 (sec)	
Pause time		8 (sec)	
Molding cycle time		40 (sec)	

As a molding example, Photograph 2.10 shows the flowing conditions of a turbocharger rotor inside the metal mold. Table 2.7 shows its molding conditions.

8. Degreasing

The degreasing process removes organic binders that have been added to retain the molding shape of powders.

For the case of a liquid, or the diffusion, volatilization or sublimation of a melting system, the volume expansion is small. Therefore, speed at which the temperature rises can be increased. On the other hand, for the case of resin components, volume expansion is large due to decomposition or gasification. Accordingly, a rapid temperature increase should be avoided. Normally, it is limited to 1-10°C/hr, which takes a long time. Most binders can be degreased below 500°C.

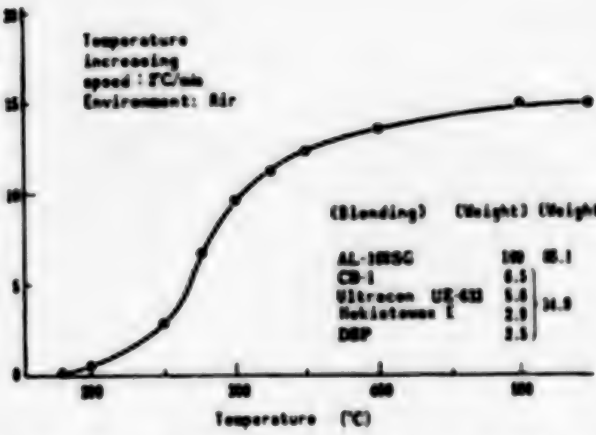


Figure 2.17 Curve Showing Decreasing Weight of Alumina Acrylic Binder

Figure 2.17 shows a reduction in volume for the alumina acrylic binder. The environments include the air, N_2 and Ar gas, depending upon the kinds of powdered raw materials. In order to shorten the processing time, the pressure degreasing method, decompression degreasing method, and super critical gas degreasing method have been adopted.

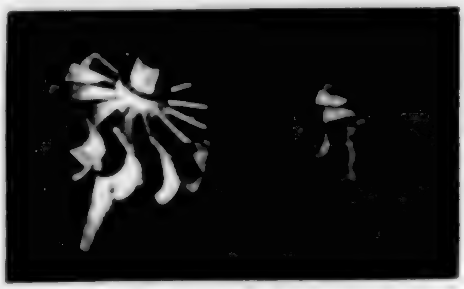
As an example of degreasing, a turbocharger rotor is discussed. Since the temperature increasing speed is held to 2-5°C/hr at normal pressure for ceramic, the process takes 60-150 hours at 100-400°C. On the other hand, it only takes about 30 hours for ferrous metal powder when the same mold is employed but the temperature is increased by 10°C/hr. However, if binder is removed completely, the material becomes brittle, making it difficult to handle. The process is usually completed at a temperature that is slightly lower than the specified one. Photograph 2.11 shows the interior of a normal pressure degreasing furnace.

Photograph 2.11 Normal Temperature Degreasing Furnace

9. Sintering

Sintering following degreasing is carried out according to the usual procedure. In order to completely remove the residual binder, the molded product is kept at 400°C for a certain amount of time. Then, the temperature is increased by 100-200°C/hr to obtain 89-90 percent of the melting point. The product is maintained at this temperature, and sintering is completed. Examples are shown by the following photographs.

Photograph 2.12 Alumina Turbine Blade (Left: molded body; Middle: degreased body; Right: sinter)



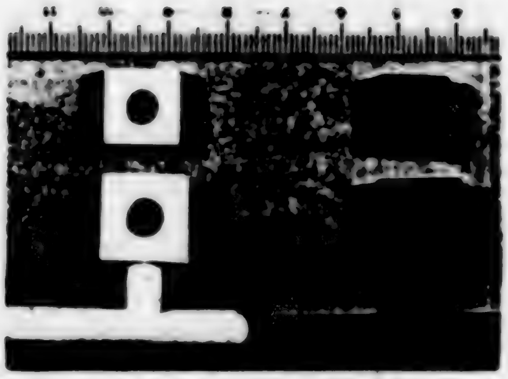
Photograph 2.13 Alumina Turbocharger Rotor (Left: green molded body; Right: sinter)

Photograph 2.12 illustrates the progress from a molded body to a sinter for a turbine blade made from an alumina compound. Almost no contraction occurs after the degreasing process. Due to contraction pressure at sintering, the material becomes more dense. Photograph 2.13 shows the molded body and sinter of a turbocharger rotor made from the same material as mentioned above. Its linear contraction coefficient is 13.8 percent in the radial direction.

Photograph 2.14 shows the molded body and sinter of a tensile/bending test piece made from a compound of Fe-Ni(8 percent) and binder 10 percent.

Photograph 2.15 shows the molded body and sinter for alumina and ferrite compounds. Figure 2.18 [not reproduced] shows the sintering conditions of a ferrite molded body.

Photograph 2.14 Fe-Ni (8%) Green Molded Body (left) and White Sinter (right)



Photograph 2.15 Alumina Molded Body (lower left), Sinter (upper left)/Ferrite Molded Body (lower right), Sinter (upper right)

In order to avoid deformation during the degreasing and sintering processes, the method burying the compound in a coarse powder of the same material is typically used.

The injection molding method for fine ceramics is the process offering the prominent and excellent features described here. Nevertheless, several problems remain to be solved. It is strongly desired that the application products taking these problems into consideration, and also that the problems associated with blending and processes be improved.

20149/9365

Trends in Optoelectronics Industry in Japan

43064060 Tokyo PROCEEDINGS OF 1ST MEETING ON LIGHTWAVE SENSING TECHNOLOGY in Japanese 23, 24 Jun 88 pp 89-95

[Article by S. Ishihara and M. Isumaki, Optoelectronics Industry and Technology Development Association]

[Text] **Abstract**

The "optoelectronics industry," which includes such diverse fields as "optical communications," "optical memory," "optical sensing," "optical energy application," etc., has continued to sustain a growth rate of more than 10 percent over the past several years, with the scale of production in FY87 is estimated to reach about ¥1.23 trillion. Following is a summary of the results of a survey and analysis of the current status (FY87) and an outlook (for FY92) of the optoelectronics industry, emphasizing production and demand in Japan.

I. Introduction

The optoelectronics industry has not only been growing within the industry itself, but has also been contributing significantly to the advancement of frontier industries, the realization of a highly advanced information society, and the realization of a high standard of living in Japan through its support of the growth in the electronics, information, and energy industries.

The Optoelectronic Industry and Technology Development Association has been conducting annual surveys using a questionnaire on the "production scale of the optoelectronics industry (on a monetary basis)" since FY80. In the survey, the products related to the optoelectronics industry are subdivided into 28 survey items in the categories of optical components, optical equipment and apparatus, and optical applied systems to determine the production scales of these items in Japan. The results of the survey are summarized in Table 1.

The total production of the optoelectronics industry in FY87, in monetary terms, increased about 18 percent over the production during the preceding fiscal year, reaching about ¥1.23 trillion. Although the growth rate decreased from that of the preceding fiscal year (about 23 percent) due to expansion of the production scale, it has been maintained at a high level.

Table 1. Production Scale of Optoelectronics Industry

Unit: million yen. Numbers in () show the percentage relative to the preceding fiscal year

	Name of product	Actual production for FY85	Actual production for FY86	Estimated production for FY87
Optical component	Light emitting element	134,719(110.6)	12,994(91.3)	132,132(107.4)
	Semiconductor laser	40,797	39,459	41,242
	Gas laser	8,027	7,614	7,586
	Solid-state laser	3,635	2,920	2,813
	Light emitting diode	82,198	72,930	80,400
	(For communications)	(5,075)	(5,101)	(6,648)
	(For noncommunications use)	(77,123)	(67,829)	(73,753)
	Other lasers	625	71	90
	Light receiving element (separate light receiving element, etc.)	23,946(109.1)	39,600(165.4)	48,195(121.7)
	Composite optical element	32,463(82.6)	35,159(108.3)	40,604(115.5)
	Solar cell	10,565(116.1)	10,094(95.5)	9,900(98.0)
	Optical fiber (including cable)	54,059(107.0)	70,635(130.7)	73,286(103.8)
	Quartz fiber	51,771	67,710	69,911
	Nonquartz fiber (plastic light guide, etc.)	2,288	2,925	3,375
	Other optical components	46,371(128.6)	60,226(129.9)	62,631(104.0)
	Connector and plug	6,933	9,057	11,408
	Optical circuit component, etc.	39,438	51,169	51,223
	Subtotal	302,123(108.4)	338,708(112.1)	336,748(108.2)
Optical equipment and apparatus	Optical transmission equipment and apparatus	28,234(88.6)	51,577(182.7)	64,586(125.2)
	Optical measuring instruments	20,717(124.1)	21,900(105.7)	26,896(122.8)
	Line laying equipment	5,024(108.0)	5,726(114.0)	6,944(121.3)
	Sensor utilizing optical fiber	2,501(182.8)	4,124(164.9)	5,257(127.5)
	Sensor utilizing disk	6,894(98.6)	9,091(131.9)	10,522(115.7)
	Optical disk	249,653(217.4)	321,473(128.8)	364,276(113.3)
	Compact disk device	198,252	257,867	274,048
	Video disk device for reproducing only	45,783	49,500	57,826
	DRAW-type optical disk device (document and data files	5,130	10,585	22,959
	Recording medium (for CD-ROM and DRAW)	488	3,521	9,443

[continued]

[Continuation of Table 1)

	Name of product	Actual production for FY85	Actual production for FY86	Estimated production for FY87
[continuation]	Optical input/output device	75,943(414.2)	95,020(125.1)	125,264(131.8)
	Medical laser equipment	4,833(112.1)	4,568(94.5)	4,882(106.9)
	Production equipment utilizing laser, etc.	52,878(108.3)	68,249(129.1)	98,591(144.5)
	Subtotal	446,677(157.7)	581,728(130.2)	707,218(121.6)
Applied optical system	Optical communications system	92,662(126.5)	115,629(124.8)	147,016(127.1)
	Public communications system	53,154	64,935	82,393
	Communications system for specific user	39,508	50,694	64,623
	Others	6,434(93.7)	6,247(97.1)	7,353(117.7)
	Subtotal	99,096(123.7)	121,876(123.0)	154,369(126.7)
	Total of optoelectronics products	847,896(132.0)	1,042,312(122.9)	1,228,355(117.8)

It has reached a level 15 times larger than the ¥80 billion of FY80 when the survey was initiated, which corresponds to an average annual growth rate of about 50 percent (about 35 percent during the past 5 years), representing a rapid growth not seen in other industries. Examining the breakdown of the data for FY87, it is seen from Table 1 that the optical component category, which includes lasers and optical fibers (including cables), represents ¥366.7 billion (component ratio of 30 percent), the optical equipment and apparatus category, which includes optical disks and optical input/output equipment, represents ¥707.2 billion (57 percent), and the optical applied system category, which includes optical communications systems, represents ¥154.4 billion (13 percent).

Reclassifying the survey results into the fields of communications, information, and energy, the communications field represents ¥379.6 billion (component ratio of 30.9 percent), the information field ¥727.3 billion (59.2 percent), and the energy field ¥121.4 billion (9.9 percent), with the ratios among the three fields remaining approximately constant over the past 3 years. Changes, in monetary terms, of production since FY81 are shown in Figure 1.

As trends during the past several years, a decrease in the share of optical components and an increase in the share of optical equipment and apparatus, have been observed in the optoelectronics industry as a whole. These trends still persist in FY87. As a cause for the increase in the share of optical

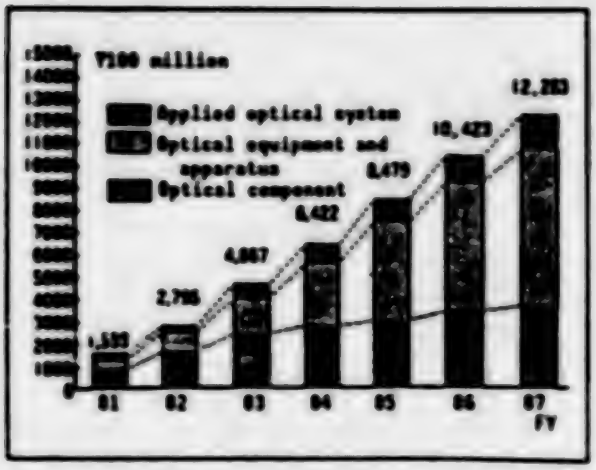


Figure 1. Changes in Domestic Production Scale of Optoelectronics Industry

equipment, one may mention the fact that the production scale of optical disks (compact disk devices, video disk devices used exclusively for reproducing, and direct-read-after-write (DRAW) type optical disk devices) is remaining large. In FY87 it reached ¥350 billion and, together with the optical communications systems, is leading the expansion of the optoelectronics industry market. On the other hand, as for the optical components, due to a sudden decrease in the cost of optical fibers, semiconductor lasers, etc., which have been contributing substantially to the expansion of the scale of optical component production, its growth in monetary terms is suffering from stagnation in spite of its increase in production volume. As a result, its share of the total optoelectronics industry in FY87 decreased from about 33 percent to about 30 percent. However, the need for optical components demanded in large volume, such as those for light-emitting diodes for short distance optical communications systems and two-dimensional array sensors for solid-state cameras, has been expanding rapidly, indicating a trend which may even cancel out the effect of the price drop of optical fibers, semiconductor lasers, etc.

II. Details of Production Scale of Optoelectronics Industry in FY87

A breakdown of the optoelectronics industry scale of ¥..23 trillion will now be given for each category, i.e., optical components, optical equipment and apparatus, and optical applied systems.

1. Optical Components

Optical components production in FY87 amounted to ¥366.7 billion, with its growth rate remaining at only 8 percent relative to that of the previous year (an increase of about ¥28 billion). The reason for the growth rate remaining at such a low level is, as mentioned above, due to the rapid drop in the price of optical fibers, semiconductor lasers, etc., so that in spite of an increase in the volume of production, the growth rate on a monetary basis was at a low level. As can be seen from Figure 2, a breakdown of the principal optical components shows: ¥84 billion (component ratio of 22

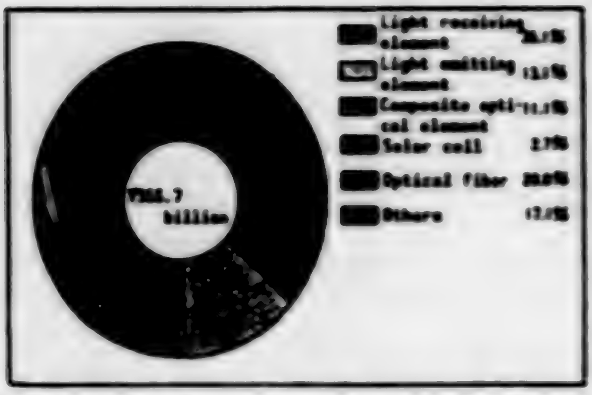


Figure 2. Breakdown of Production of Optical Components in FY87

percent) for light-emitting diodes, ¥73.3 billion (20 percent) for optical fibers, ¥48.2 billion (13 percent) for light-receiving elements, ¥41.2 billion (11 percent) for semiconductor lasers, and ¥40.6 billion (11 percent) for composite optical elements.

The growth rate for the semiconductor lasers remained at 4 percent over the preceding year, which was smaller than that of the optical components as a whole. The price drop mentioned earlier is making rapid progress, not only for the semiconductor lasers for CD use, but also for the semiconductor lasers for long wavelength communications systems. Since the frequent use of light emitting diodes as light-emitting elements is anticipated in optical communications systems, which are expected to grow in the future, the current trend involving the growth rate of semiconductor lasers is expected to persist for the time being until the appearance of new products, such as high power semiconductor lasers.

Both the gas laser and the solid-state laser recorded negative growth. For the gas laser field, the start of production of the excimer laser or production expansion is anticipated, as is the semiconductor-laser-excited solid-state laser in the solid-state laser field.

The light emitting diode showed a growth rate of 10 percent over the preceding year, which is larger than that of the semiconductor laser. In particular, the growth of diodes for communications use was as large as 30 percent. The amount of production for the light emitting diodes as a whole exceeded ¥8[illegible].

The FY87 production scale of optical fibers reached about ¥73 billion, but the growth rate, relative to that of the previous year, dropped, remaining at 4 percent. The growth rate of the single mode fiber, which occupies the largest share of the optical fibers under production, was low at 2 percent relative to the preceding year, strongly reflecting the influence of the price drop. On the other hand, the growth rate of optical fibers other than the quartz-based fiber was high at 15 percent. Such a difference between the quartz-based and nonquartz-based fibers is expected to persist for some time in the future.

The optical connector and plug showed the high growth rate of 26 percent over the preceding year, exceeding the ¥10 billion level and rising to ¥11.4 billion. As a foundation for this trend, it may be pointed out that an increase in the demand for a product which accompanied the growth of the optical transmission equipment and the growth of the optical communications systems, which do not contribute appreciably to the demand for semiconductors or quartz-based optical fibers, served as factors for the demand increase for optical connectors and plugs.

2. Optical Equipment and Apparatus

The production sum of the optical equipment and apparatus in FY87 was ¥707.2 billion, showing an increase of 22 percent (about ¥126 billion) over the preceding year.

As shown in Figure 3, a breakdown of the optical equipment and apparatus is as follows: ¥274 billion (component ratio of 39 percent) for compact disks, ¥57.8 billion (8 percent) for video disks for reproducing only, ¥125.3 billion (18 percent) for optical input/output devices such as the laser printer, and ¥98.6 billion 14 percent) for laser processing devices, etc.

Figure 3. Breakdown of Production Total of Optical Equipment and Apparatus in FY87

Production of the compact disk had been expanding in scale, with a high growth rate, up to the past fiscal year, but the growth for the current fiscal year has remained at only 6 percent over the preceding year. A price drop has also been continuing for this product. However, the overseas production is also expanding and, as a result, it is believed that the growth rate is at a lower level than previously. On the other hand, the DRAW-type optical disk, which last year showed a high growth rate of 135 percent over the preceding year, continued its growth this year with a ratio of 117 percent over last year, exceeding ¥20 billion. Furthermore, the video disk for reproducing only showed an increase of 17 percent, which is greater than last year's growth rate of 10 percent over the preceding year. As a basis for this, the expanding demand in the United States in particular is believed to be contributing significantly.

Optical transmission equipment, which last fiscal year exhibited a production scale of ¥58 billion, exceeding the ¥50 billion level, and a high growth rate of 29 percent over the preceding year, this year experienced a high growth rate of 25 percent, which exceeds that of optical equipment and apparatus. Although expansion in terms of the amount of equipment for short-distance optical communications systems has been continuing, as mentioned earlier, the cause for such high growth in monetary terms is credited to the increase in general transmission equipment with high unit prices. It is conjectured that investments in updating information systems, etc., in financial organizations, including those of foreign capital enterprises, are influencing the trend.

The optical input/output equipment also showed high growth, reaching 32 percent relative to the preceding year. In production terms, the increase was at about ¥30 billion. Such growth seems to be influenced by the expansion of the semiconductor laser printer. The semiconductor laser printer has been experiencing, along with a rapid decrease in price, a fast demand expansion due to this price drop, and it is securing its position among the high class printers in the OA field where diversified products coexist with those of the optical type.

As mentioned earlier in conjunction with the optical components, the growth rates for the gas laser and the solid-state laser have both been negative. Processing equipment utilizing lasers is included in the figure listed above for laser processing equipment etc., while a growth rate of about 13 percent is estimated for the carbon dioxide laser applied equipment. That is, for a single laser, the production scale showed negative growth along with a price drop. For the equipment, however, a different trend from that of the single laser has been observed due to the price increase accompanying the improved equipment functions and the expansion of the demand fields.

3. Optical Applied System

The production sum for optical applied systems for FY87 was ¥154.4 billion showing an increase of 28 percent (about ¥32.5 billion). Its breakdown, as shown in Figure 4, is: ¥147 billion (component ratio of 95 percent) for optical communications systems, and ¥7.4 billion (5 percent) for other systems.

Figure 4. Breakdown of Production Sum of Applied Optical Systems for FY87

Of these, the breakdown of the production scale for optical communications systems was ¥82.4 billion for the public communications systems and ¥64.6 billion for communications systems for specific consumers. The growth rate relative to the preceding year for both communications systems was 27 percent, higher than the growth rate for the optoelectronics industry as a whole and, as a result, its share in the optoelectronics industry increased. As mentioned earlier in conjunction with the optical equipment and apparatus, the demand for optical communications systems is increasing steadily, the result of which is reflected in this high growth rate.

III. Outlook (5 Years Hence) for Optoelectronics Industry

Following are the results of an estimate for the future, 5 years from now, based on the present status of the optoelectronics industry as described in the preceding sections. In preparing the estimate, the production volume was calculated from the production amount, and the domestic need was determined from the domestic production by taking the export ratio into consideration. The optical product used as the objects of the estimate were basically the same as those for the production statistics for FY87. However, for such products as "applied optical systems" and "semiconductor lasers, etc.," the average scale per system and its usage were not clearly definable, making them difficult to estimate, so they were excluded from the project. In other words, the production aspect was given priority over the demand aspect, and only those existing products offering clear images were adopted as objects to be estimated.

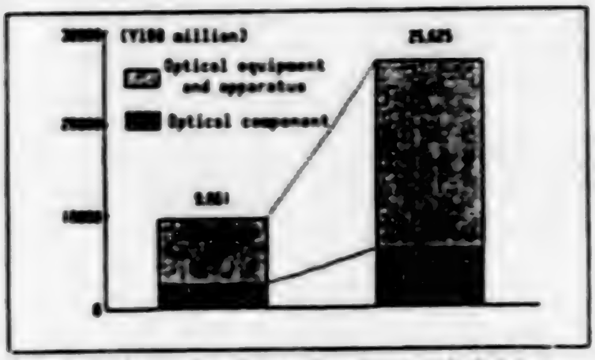
The results of the estimates for the current fiscal year are shown in Table 2.

As will be described shortly, since some optoelectronic products were not adopted as objects of the estimate but were included among the optoelectronics products used for the production statistics for FY87, the production scales for FY87 shown in the following figures have been assigned smaller values than were shown above. The changes in the production sum for the optoelectronic products adopted as the objects of the estimates are shown in Figure 5. As shown in Table 2, for the Japanese injection molding method in 1992, the sum of the optical parts and optical equipment and apparatus has been estimated at ¥2.56 trillion, and can be compared to the ¥906 billion for FY87. The average annual growth rate of domestic products for this period is estimated to be 23 percent, which is approximately the same as the average annual growth rate (24 percent) for the period 1984-87. The reason for obtaining an estimate for which the growth rate is approximately equal to the existing rate is that the "optical disk," which has the maximum component ratio, should be able to maintain its high growth rate through an expansion of its functions (from ROM to WORM and ERASABLE), as well as due to the commercialization of compound products (CV-D, CV-I, and CD radio cassettes).

However, the domestic demand came out with a low growth rate of 19.3 percent, which is approximately the same as that for FY87 relative to the preceding year. As a result, as seen from the product group shown in

Table 2. Results of Estimates for Optoelectronics Industry (Summary)

Item		Amount of production (¥ million) FY87	Amount of production (¥ million) FY92	Amount of production (¥ million) FY87	Amount of production (¥ million) FY92	Growth rate Production 92/87	Growth rate Demand 92/87
1.	Semiconductor laser	34,500	69,500	21,640	40,600	15.04	13.42
2.	Gas lasers	6,700	17,100	6,700	16,000	20.61	19.01
3.	Solid-state lasers	2,500	9,600	3,220	13,320	30.88	32.83
4.	Light emitting diodes	61,900	99,440	44,070	71,100	9.94	10.04
5.	Separate light receiving elements	21,750	46,200	16,400	33,090	16.26	14.94
6.	Array-type light-receiving elements	26,000	52,000	22,750	46,800	14.87	15.52
7.	Composite optical elements	41,000	69,000	41,000	59,920	10.97	7.88
8.	Solar cells	9,900	16,300	6,990	10,490	10.49	8.45
9.	Optical fibers	68,710	85,420	47,510	67,890	4.45	7.40
10.	Bundle fibers	2,400	10,000	2,400	9,000	33.03	30.26
11.	Passive optical components	15,850	50,200	14,200	36,450	25.93	20.75
Subtotal for optical components		291,210	524,760	226,880	404,664	12.50	12.26
12.	Optical transmission equipment and apparatus	62,950	156,200	59,110	139,380	19.93	18.72
13.	Measuring instruments for optical transmission	13,510	42,670	11,360	27,810	25.86	19.62
14.	Equipment for line laying	5,000	10,000	2,500	5,000	14.87	14.87
15.	Optical sensors sensor utilizing laser or fiber)	5,950	20,850	5,950	17,910	28.50	24.66
16.	Optical disk players	351,000	1,189,100	119,660	444,100	27.64	29.99
17.	Optical printers	110,670	419,000	54,660	133,200	30.51	19.50
18.	Laser and LED scanners (bar code reader)	10,900	28,000	10,900	28,000	20.77	20.77
19.	Medical laser equipment	4,830	19,900	3,310	11,620	32.73	28.58
20.	Production equipment utilizing laser	36,080	131,000	36,080	82,700	29.42	18.05
21.	Equipment for printing and printing plate preparation	14,000	21,000	11,200	16,800	8.45	8.45
Subtotal for optical equipment and apparatus		614,890	2,037,720	314,730	906,520	27.08	23.56
Total		906,100	2,562,480	541,610	1,311,180	23.11	19.34



Note: Applied optical systems and products not included as objects of estimation have been excluded.

Figure 5. Change in Production Sums of Optical Components and Optical Equipment and Apparatus

Table 2, dependence on the overseas market will be increased to 48.8 percent for FY92, compared with the 40.3 percent for FY87.

As mentioned earlier, the domestic products for FY87 included in the optical products which are the objects of the estimate totaled less than had been anticipated for FY87, but this included the optical products that were excluded from the estimate. More specifically, the sum of production scales of the products adopted as the objects for the estimates was smaller, than that for FY87, by about ¥180 billion for optical components and the optical equipment and apparatus combined, and by about ¥330 billion for the total, including the applied optical systems that were excluded from the estimate. Accordingly, in order to obtain a production scale for FY92 that will correspond to that for FY87, it will be necessary to make a correction by taking the above difference into consideration.

With the correction for the products excluded from the estimate, about ¥910 billion (¥200 billion for optical components, ¥320 billion for optical equipment and apparatus, and ¥390 billion for systems) is to be added to the production scale. As a result, the domestic production scale for FY92 will amount to about ¥3.47 trillion.

Here, the correction of ¥200 billion for FY92 includes ¥100 billion which is anticipated for minute optical parts, etc., that are among miscellaneous optical products. The above figure also includes an estimated produ- ion scale of about ¥100 billion or miscellaneous semiconductor lasers.

Furthermore, the corrected sum of ¥320 billion for the optical equipment and apparatus includes an estimated production expansion for such fields as optical fiber utilization and sensors which utilize lasers in addition to ¥300 billion for optical disks.

According to the corrected estimated value, which takes into account the abovementioned correction figure, the average annual growth rate for that 5-year period will be 23 percent. However, this result does not take into account the subscriber-oriented communications system, solid-state cameras, displays, etc. Therefore, on the basis of the estimated result, the

possibility arises of augmenting the results with ¥400 billion for subscriber-oriented communications systems, ¥1 trillion for solid-state cameras, and ¥400 billion for displays.

The production sum for the optical components adopted as the object of the estimate was about ¥290 billion for FY87, and the production estimate for the identical optical products for FY92 was estimated at ¥520 billion. The average annual growth rate for the period will come out then as 12 percent. The breakdown of the production sum for FY92 is shown in Figure 6.

Figure 6. Breakdown of Production Sum of Estimated Amounts for Optical Components (FY92)

As shown in Figure 6, the highest share of domestic production for FY92 is 37 percent, for light-emitting elements, followed by 19 percent for light-receiving elements, 18 percent for optical fibers, 13 percent for composite optical elements, 10 percent for passive optical components, and 3 percent for solar cells. The light-emitting elements, light-receiving elements, optical fibers, etc., all represent optical components that have experienced rapid price drops. However, the device experiencing the most developmental problems is the light-emitting element, followed by the light-receiving element. This aspect is reflected in the above ratio. (Needless to say, when optical fibers are introduced to subscriber-oriented systems, it is conceivable that the significance of optical fibers will maintain its present status or increased slightly.)

Namely, since it is possible to develop new products which can cancel a rapid price drop, price decreases affect the amount of production amount less directly than does the appearance of optical fibers, etc., which comprise a large portion of the component ratio.

The production sum of the portion of the optical equipment and apparatus which was adopted for the estimate was about ¥610 billion for FY87, and the production scale of the same products for FY92 has been estimated at about ¥2 trillion. The average annual growth rate for the period emerged as 27 percent, which is more than twice that for optical parts previously.

The breakdown of the optical equipment and apparatus for FY92 is shown in Figure 7.

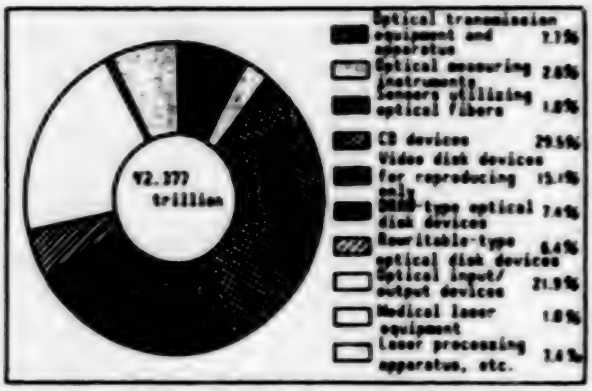


Figure 7. Breakdown of Estimated Production for Optical Equipment and Apparatus (FY92)

The highest production rate among optical equipment and apparatus is claimed by the compact disk device, representing 30 percent. Following is 22 percent for the optical input/output device, 15 percent for video disks for reproducing only, 8 percent for optical equipment and apparatus, 7 percent for laser processing devices, and DRAW-type optical disk devices, 6 percent for rewritable-type optical disks, 3 percent for optical measuring instruments, and 1 percent for sensors utilizing optical fibers and medical laser devices. The optical disk-related devices occupy 58 percent, representing more than half of the total production of optical equipment and apparatus for FY92. In the optical disk-related field, during the next 5 years the development of various new products is anticipated, e.g., a player which can also reproduce other optical disk media. In the present estimate, products related to CD-ROM, such as CD-I and CD-V, are included in the CD category.

Also, in the estimate for the current fiscal year, it was not assumed that the subscriber-oriented systems would be put into practical use. That is, although it is believed that subscriber-oriented optical communications systems will surely enter practical use, and that when this happens, a demand of several hundred billion yen annually will be created, the exact time of this occurrence is as yet unknown. Accordingly, the subscriber-oriented systems were excluded from the objects of the estimate for the current fiscal year with the provision that be taken into account when their realization becomes definite.

IV. Optoelectronization Index

The optoelectronization index is an index aimed at a quantitative recognition of the optoelectronization factor (a measure representing the degree of popularization of optoelectronic products in a specified field).

The optoelectronization indices announced last fiscal year were adopted by adding new data acquired during the past year. The principal optoelectronization indices will be described below.

1. Optical Transmission Equipment

In this area, the optoelectronization index is defined as the ratio of the optical communications equipment to the wired communications carrier equipment. Allowing for the fact that fields will remain for which electrical communications will be advantageous, even in the future, the upper limit of the popularization factor is set at 50 percent. It should be noted that the reason for the gradual change of the index from one fiscal year to the next in comparison to that of the optoelectronization factor for optical fibers is that electronic equipment, such as multiplexing equipment, is being used in the optical communications systems. The computation results of the index are shown in Figure 8.

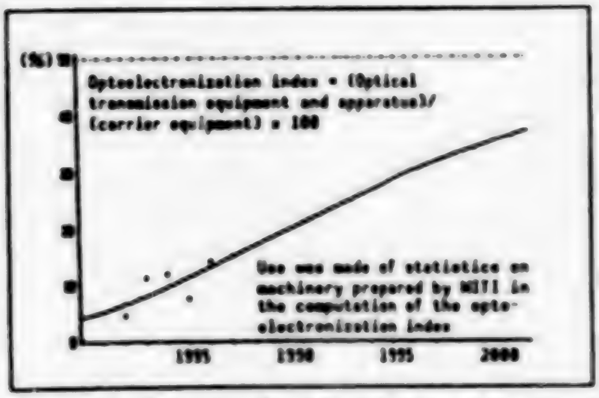


Figure 8. Optoelectronization Index for Optical Transmission Equipment

2. CD Player

The popularization of CD players is progressing at a rapid pace, and it is believed that it will eventually drive away analog players. That is, the upper limit of the index is set at 100 percent. The results of the computation are shown in Figure 9.

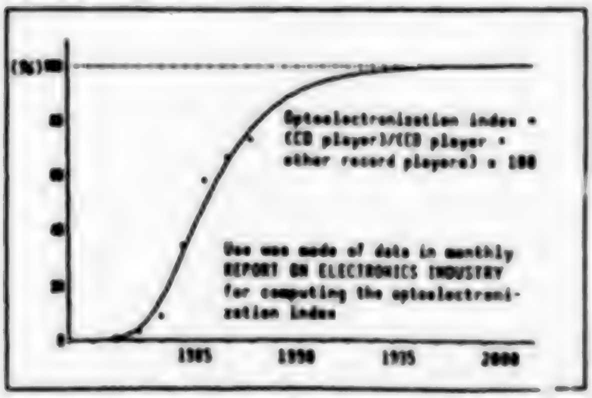


Figure 9. Optoelectronization Index for Number of CD Players

3. Industrial Lasers

Industrial lasers have been compared with optical equipment and instruments. The optical instruments include cameras, telescopes, etc., so that the upper limit of the index has been set at 10 percent. The results of the computation are shown in Figure 10.

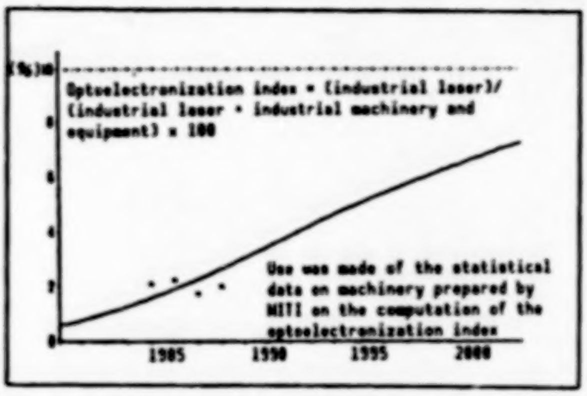


Figure 10. Optoelectronization Index for Industrial Lasers

4. Optical Disks

Since the existing electrical wires include not only those used for communication purposes but also those for power transmission, use was made here of the data for the existing electrical wires. The results of the computation are shown in Figure 11.

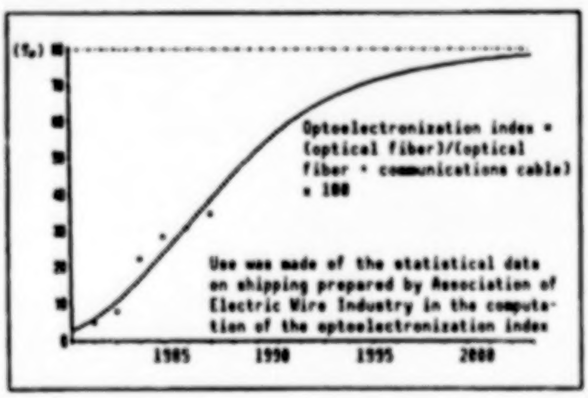


Figure 11. Optoelectronization Index for Optical Fibers

5. GNP

The ultimate goal of the ratio of the injection molding method to the GNP was set at percent. In order to obtain the proper ratio of the production sum of the injection molding method, calculations avoided overlaps among the component parts, equipment, and systems. The calculated results are shown in Figure 12.

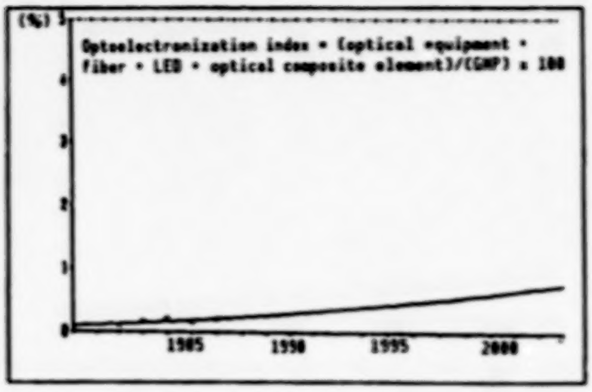


Figure 12. Optoelectronization Index Relative to GNP

V. Conclusion

The optoelectronics industry, which has seen rapid growth as a typical high technological industry, attained the production sum of ¥1.23 trillion, with some optoelectronics products moving toward stabilized growth. Of these products, although countermeasures are needed for trade problems involving technologically advanced countries and for catch-up problems of the NICS, the need to actively promote the development of more highly advanced technologies and the development of new products is increasing in order for the injection molding method to realize continuous growth.

Now that Japan's domestic as well as international responsibilities are increasing in significance due to the expansion of her industrial scale, it may be said that the necessity for constructing guiding policies, based on thorough examination, for the future of Japanese injection molding method and for Japan's international role is increasing.

20121/9365 END

Foreign Broadcast Information Service (FBIS) and Joint Publications Research Service (JPRS) publications contain political, economic, military, and sociological news, commentary, and other information, as well as scientific and technical data and reports. All information has been obtained from foreign radio and television broadcasts, news agency transmissions, newspapers, books, and periodicals. Items generally are processed from the first or best available source; it should not be inferred that they have been disseminated only in the medium, in the language, or to the area indicated. Items from foreign language sources are translated; those from English-language sources are transcribed, with personal and place names rendered in accordance with FBIS transliteration style.

Headlines, editorial reports, and material enclosed in brackets [] are supplied by FBIS/JPRS. Processing indicators such as [Text] or [Excerpts] in the first line of each item indicate how the information was processed from the original. Unfamiliar names rendered phonetically are enclosed in parentheses. Words or names preceded by a question mark and enclosed in parentheses were not clear from the original source but have been supplied as appropriate to the context. Other unattributed parenthetical notes within the body of an item originate with the source. Times within items are as given by the source. Passages in boldface or italics are as published.

SUBSCRIPTION/PROCUREMENT INFORMATION

The FBIS DAILY REPORT contains current news and information and is published Monday through Friday in eight volumes: China, East Europe, Soviet Union, East Asia, Near East & South Asia, Sub-Saharan Africa, Latin America, and West Europe. Supplements to the DAILY REPORTs may also be available periodically and will be distributed to regular DAILY REPORT subscribers. JPRS publications, which include approximately 50 regional, worldwide, and topical reports, generally contain less time-sensitive information and are published periodically.

Current DAILY REPORTs and JPRS publications are listed in *Government Reports Announcements* issued semimonthly by the National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, Virginia 22161 and the *Monthly Catalog of U.S. Government Publications* issued by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

The public may subscribe to either hardcover or microfiche versions of the DAILY REPORTs and JPRS publications through NTIS at the above address or by calling (703) 487-4630. Subscription rates will be provided by NTIS upon request. Subscriptions are available outside the United States from NTIS or appointed foreign dealers. New subscribers should expect a 30-day delay in receipt of the first issue.

U.S. Government offices may obtain subscriptions to the DAILY REPORTs or JPRS publications (hardcover or microfiche) at no charge through their sponsoring organizations. For additional information or assistance, call FBIS, (202) 338-6735,or write to P.O. Box 2604, Washington, D.C. 20013. Department of Defense consumers are required to submit requests through appropriate command validation channels to DIA, RTS-2C, Washington, D.C. 20301. (Telephone: (202) 373-3771, Autovon: 243-3771.)

Back issues or single copies of the DAILY REPORTs and JPRS publications are not available. Both the DAILY REPORTs and the JPRS publications are on file for public reference at the Library of Congress and at many Federal Depository Libraries. Reference copies may also be seen at many public and university libraries throughout the United States.

END OF
FICHE
DATE FILMED
11 Jan 89